VOL. 80 No. 2039
AUGUST 9 1958

Telephone: FLEet Street 3212 (26 lines)
Telegrams: Allangas - Fleet - London

CHEMICAL AGE

BOUVERIE HOUSE · 154 FLEET STREET · LONDON · EC4

Editor M. C. HYDE
Manager H. A. WILLMOTT
Director N. B. LIVINGSTONE WALLACE

Midland Office
Daimler House, Paradise Street, Birmingham. [*Midland* 0784-5]

Leeds Office
Permanent House, The Headrow, Leeds 1. [*Leeds* 22601]

Scottish Office
116 Hope Street, Glasgow C2. [*Central* 3954-5]

IN THIS ISSUE

Acrylonitrile via Ethylene	218
Pest Infestation Report	219
ICI's New Nitroglycerine Plant	220
Sulphuric Acid Production	220
High Polymer Conference—3	221
DSIR	223
Distillates	224
Indian News Letter	225
Knapsack's Acrylonitrile Process	226
Overseas News	227
Equipment Review	229
Bookshelf	231
Gypsum and Anhydrite	233
Dollar Import licensing	233
Chemical Pioneers	234
People	235
Commercial	236
Trade Notes	237
Market Reports	237
Patents	238

Annual subscription is: home, 52s 6d, overseas, 60s, single copies 1s 6d (by post 1s 9d)

THE APPLEBY PROCESS

FURTHER details have now been released of the construction and operation of the new combined desulphurising and sulphuric acid pilot plant developed by Appleby-Frodingham Steel Co., Scunthorpe (see CHEMICAL AGE, 17 May, p. 912). It will be recalled that the large pilot plant, which purifies up to 3 million cu. ft. of coke oven gas per day, consists essentially of a fluidised hot iron oxide absorber and regenerator, is capable of removing hydrogen sulphide and organic sulphur to very low levels; at the same time sulphuric acid in considerable quantity can be recovered. The quantity of sulphur gases available from the full size plant is expected to suffice for the production of more than 250 tons of sulphuric acid monohydrate per week. The gases from the regeneration are particularly suitable for use in a contact sulphuric acid plant, since they are dry and are free from arsenic, chlorides and oxides of nitrogen.

Other claims made for the new process (British Patent 719,056; other related patents are pending) are that, as it employs hot gases, it is much faster than the conventional iron oxide boxes or purification towers. Use of fluidised beds reduces the amount of spent oxide by four-fifths and simplifies its handling and distribution. In compactness and capital cost, the process is stated to compare very favourably with existing methods, while the entire operation of the full-scale plant, it is estimated, is capable of being handled by two men per shift.

This fluidised iron-oxide process is operated at a temperature of 350° to 400° C. The sulphided ore is conveyed to a regenerator chamber where it is oxidised in a current of air at 600° to 800° C., the exit gases being suitable for sulphuric acid manufacture. No oxygen is added to the incoming crude gas. Regenerated oxide is returned to the absorber, where it provides one-third of the heat requirements of the process, the remainder being obtained by heat exchange of the cold incoming crude gas against outgoing hot desulphurised gas in a suitable heat exchanger.

Over 95 per cent sulphur absorption has been achieved from gas containing 600 grains of H_2S per 100 cu. ft. in conjunction with the removal of about the same percentage of organic sulphur. With suitable modifications, reports Dr. L. Reeve, chief metallurgist, Appleby-Frodingham Steel Co. (*J. Inst. Fuel*, 1958, **31**, 319), it is possible to raise the sulphur removal almost to 100 per cent and to exceed Gas Referee standards of purification.

The percentage desulphurisation attained in the pilot plant was found to depend on bed temperature, bed depth, rate of blowing and possibly ore sizing, although this latter has not been fully investigated.

With regard to bed depth, it was noted in early trials that the effect of increasing bed depth when using a single bed was less than linear. Checks were therefore made to see if there was any advantage in running two or more shallow beds in series rather than one deep bed. The new plant consists basically of two fluidised beds which contain Northampton ironstone.

Results obtained after maintaining a fluidised bed of ore around 300° to 390° C. indicated that with crude coke-oven gas containing approximately 1 per cent of H_2S, overall figures of percentage desulphurisation from 95 to 99 might be expected, depending upon the temperature.

Removal of organic sulphur in the process has been investigated both in the laboratory and on the pilot-plant scale. The degree of organic sulphur removal runs parallel with the percentage H_2S removal. This strongly suggests

that the hot oxide used acts as a catalyst for the conversion of organic sulphur to hydrogen sulphide which is then absorbed by the oxide.

Once the plant has started up, it is self-sustaining on temperature. The circulating ore is heated up to 250° C. by preheating units and the plant is brought up to temperature by the ore circulation and heat exchange system. Bed depths are kept under observation by reading pressures in conjunction with bed-depth-level indicators of the Tektor probe type. A radioisotope bed-level indicator is also being tried.

Operating troubles have been of minor importance. These have been caused by pressure fluctuations in the plant arising from the sudden closing of valves on the steel furnace with which the plant is connected. As safety devices therefore two pressure switches have been inserted. In the proposed full-sized plant, which will desulphurise gas for all the furnaces in the Appleby-Frodingham works (something like 32 million cu. ft. of coke-oven gas a day), these problems are likely to be reduced considerably.

As a safety measure to ensure more positive separation of the ore flowing between the regenerator and absorber inlet and exit, seals have been introduced. These seals are kept agitated by a slight gas flow, preferably of some inert gas. The exit seal also acts as a convenient means of charging fresh ore into the system during operation.

A certain degree of reduction of iron oxide takes place in conjunction with the desulphurising reaction, the extent depending upon time and temperature of operation. In the pilot plant, the ferrous iron is found to be largely re-oxidised in the regenerator, so contributing a further quota of heat to the circulating ore owing to the heat of oxidation of FeO to Fe_2O_3.

Trials have been carried out on a laboratory scale to determine the effect of regenerating the sulphided oxides by roasting in air at temperatures up to 800° C., and discovering whether such ores maintain their desulphurising activity. Some reduction in rate of reaction compared with fresh oxide has been noted, but not sufficient to affect its usefulness. Results of regeneration in the pilot plant indicate that the ore can be sulphided and regenerated many times, and still retain useful activity.

Slow breakdown of the ore takes place during the desulphurisation. Fluidising rates have been adjusted, it is reported, in such a manner that most of the fines are carried over from the regenerator and collected at one cyclone as oxidised material; the sulphided oxide collects at the other cyclone and represents only a small proportion of total fines. The amount of fines lost in this manner has been found to average about 4,000 lb. per week, during which time 20 million cu. ft. of gas are desulphurised (about 5,000 cu. ft. of gas per 1 lb. of oxide).

It is of interest to note that the overall improvement on furnace sulphur removal using the desulphurised gas, obtained by the Appleby-Frodingham process, over a long period has been estimated to be between 0.006 and 0.007 per cent of sulphur. Needless to say there has been a corresponding improvement of sulphurs in the slag, and details regarding this are to be reported in due course.

ACRYLONITRILE VIA ETHYLENE

BEHIND the development of the new Knapsack process (see page 226) have been careful economic considerations. Acrylonitrile can be produced industrially today by two different processes, both of which are based on hydrocyanic acid. Older of the two processes is that using ethylene oxide and hydrocyanic acid, which was first started in the U.S. because of the availability of large quantities of ethylene from refinery gases. Ethylene cyanhydrin, easily produced from ethylene oxide and hydrocyanic acid, is converted by dehydration into high-purity acrylonitrile, the yield being 94 per cent (US Patent No. 2, 461,492, 1949).

The process in ethylene oxide has, in general (even in the US) proved too expensive compared with direct production of acrylonitrile from acetylene (Farbenfabriken Bayer AG process) Thus today, it is estimated, not more than 20 per cent of US production is based on ethylene oxide, while the greater part is made from acetylene. The US figures are believed to be characteristic of the trend elsewhere, and it is expected that in the future acetylene will be virtually the sole raw material basis in the US.

The process for the production of acrylonitrile from acetylene and hydrocyanic acid as seen from the equation ($H_2 + HCN \rightarrow CH_2 = CH - CN$ would appear to be particularly elegant and simple. Several secondary reactions take place, however, in addition to the main reaction, so that in the most favourable case about 80 per cent of the acetylene and about 85 per cent of the hydrocyanic acid are returned as output. Also owing to the numerous by-products produced, the acrylonitrile obtained contains many impurities which can only be separated from it with difficulty, due to the close boiling points of acrylonitrile, divinyl acetylene and methyl vinyl ketone. Even a low content of these mostly unsaturated compounds interferes with polymerisation of the monomer and impairs the quality of the polyacrylonitrile.

If acrylonitrile is produced using acetylene as starting material, large investment is required to undertake acetelene production. Surplus capacities for making acetylene either from carbide or from natural gas (Sachsse process) do not exist outside the US.

Geographic limitations are also involved, as transport of acetylene is impossible and production depends on the availability of cheap electricity or cheap natural gas.

Knapsack calculate that the detour via acetaldehyde rather than use of acetylene for the direct synthesis of acrylonitrile could be profitable only if the additional output of acrylonitrile thus achieved exceeds the extra cost of making acetaldehyde from acetylene. Sennewald and Steil state that given very large outputs (about 40,000 tons a year) on the basis of acetaldehyde, the detour would be profitable as the price of acetaldehyde in that case amounts to no more than 65 per cent of the acetylene price. The raw material cost of acrylonitrile from acetaldehyde is as much as 4 per cent below that of an equal amount of acrylonitrile produced direct from acetylene.

These workers go so far as to state that the economic advance ensuing in future from the possibility of starting from acetaldehyde, instead of acetylene, in the production of acrylonitrile will be fully appreciated only if it is realised that acetaldehyde production from acetylene is in many countries today an economically outdated process. Cheap ethylene or else direct oxidation of low-boiling hydrocarbons can provide considerably cheaper acetaldehyde in these countries. This holds good in particular for the USA and it is likely to extend to all industrial countries.

Advantages of the Knapsack process for producing acrylonitrile from acetylene are, economically, first that the process gives considerably better yields, and secondly, that it starts from a raw material in which 2C + 2H are much cheaper than in acetylene. Two factors which are decisive for profitability are: investment, expenditure and operating cost; and raw material costs which depend on the prices of the raw materials used and on yields.

The Knapsack workers have made fairly precise calculations and reached the conclusion that the investment required for the acetaldehyde-hydrocyanic acid process is about 10 per cent below that for the acetylene-hydrocyanic acid process, while operating costs are the same. Raw material costs of the new process are about 20 per cent lower than those of the acetylene-prussic acid process.

PEST INFESTATION RESEARCH

PIRB Report Indicates Good Progress in Insecticidal Efficacy Studies

DEVELOPMENT of resistance by insects to insecticides is described as an 'extremely serious problem' by the Pest Infestation Research Board in its annual report *Pest Infestation Research 1957* (published for DSIR by HMSO, price 5s. (90 cents US), by post 5s. 5d.). The Board, which advises the Council for Scientific and Industrial Research on the Pest Infestation Laboratory's (DSIR) research programme, considers that insecticide resistance should be given a high priority. Substantial financial support has been offered to the laboratory by the World Health Organisation in aid of biochemical research on this project. Other work requiring urgent attention according to the report includes: further knowledge of the rates of development of insect infestations on various foodstuffs stored under different conditions; more efficient and safer chemical control measures; and the development of insect-proof packaging materials.

Another recommendation the board makes is that in order to control insect pests more effectively, the laboratory should extend its research to cover infestation of food storage and handling on board ships and in overseas territories. This will not be a completely new venture as for some years the laboratory has maintained close contact, through its Colonial Liaison Officer, with storage problems in many colonial territories.

Determining Toxicity Loss of Pyrethrum Sprays

Insecticides: Good progress has been made in a major item of work initiated in 1956, namely, the study of the insecticidal efficacy of various water-dispersible powders against eight species of beetle. This study is almost complete and the emphasis is now shifting to the determination of the persistence of these preparations.

With regard to spraying and dusting of insecticides, tests have been carried out to determine whether the heat of the propellent exhaust gases of a portable, thermal fog generator caused loss in toxicity of pyrethrum sprays. Although the pyrethrins have been exposed momentarily to temperatures of up to 880°C, biological tests against house flies, using material recovered after fogging, showed that a negligible loss of toxicity had resulted.

A comparison of deposits of malathion and lindane water-dispersible powders on filter paper and on jute sacking has shown that malathion retains its effectiveness during storage for two months at 25°C and 70 per cent RH on these substrates, whereas the effectiveness of lindane markedly decreases. The loss of toxicity of lindane is believed to be due to volatilisation. Earlier works had indicated that malathion applied as a water-dispersible powder to brick was no more persistent than lindane, but this loss of toxicity of films of malathion on bricks is now considered to be due to breakdown of the insecticide on the alkaline bricks.

Results of mixing malathion dust with grain have confirmed that 4 to 8 p.p.m. malathion should effectively protect wheat against several important species. The potentialities of methoxychlor dust for the control of certain grain-infesting beetles have been investigated. Methoxychlor is less toxic to insects than DDT and is also considerably less toxic to mammals, it might therefore have advantages in relation to human safety. Methoxychlor at 100 p.p.m. failed to prevent breeding of grain weevil, rice weevil and Cigarette Beetle, but at 50 to 100 p.p.m. it prevented breeding of the flour beetles and Lesser Grain Borer. The high doses required of this insecticide seem to rule out its practical use in being mixed with grain for insect control.

Effect of solvent on insecticidal activity has been explored. DDT at 25°C was found to be twice as toxic when applied in a non-volatile technical white oil as when applied in petroleum ether (boiling range 100° to 120°C). Applied in petroleum ether containing 7 per cent of the oil, DDT toxicity was nearly as great as when applied in the oil alone. Pyrethrins as a direct spray and DDT applied as film and spray were much more toxic if dissolved in non-volatile oil than in dibutyl phthalate. As a film, pyrethrins were somewhat more lethal if in the oil.

Experiments initiated in 1956 to determine the effect of certain dosages of DDT and lindane dusts applied to beans, maize, rice and groundnuts have been continued. Germination tests have shown no deleterious effect of the insecticides during storage of the seeds for nine months.

Work has continued on pyrethrum synergists, a series of 1-3 : 4(methylenedioxyphenyl)-*n*-alkanes, ranging from propane to the pentadecane, being investigated. All the compounds showed some activity, but the lowest and highest members of the series were poor pyrethrum synergists. The parent compound, methylene-dioxybenzene (catechol methylene ether) was entirely inactive under similar conditions of test. Maximal activity is attained with a chain-length of 10 or 11 carbon atoms. Minimal molecular requirements for this type of synergism appear to be a methylene-dioxyphenyl nucleus, coupled with a lipoid-soluble group.

A comparison of the potencies of the methylene dioxyphenyl-alkanes with those of other simple methylene-dioxyphenyl derivatives, such as 'piperonyl butoxide', suggests that an alkyl group may not furnish the optimal molecular configuration and that a slightly more polar side-chain is required for maximum synergism. This aspect of the problem is being investigated further.

Experiments have been done to compare the activity of piperonylate of the mono-*n*-butyl ether of diethylene glycol with that of piperonyl butoxide in synergising pyrethrins. Applied as direct sprays and films in heavy oil and topically in cyclohexanone, piperonyl butoxide proved to be superior.

Application of radioisotope techniques: Development of radiometric paper chromatographic and 'labelled pool' techniques has opened up new fields of insecticidal research of both fundamental interest and potential practical value, the report states. The virtual immunity to several of the hitherto most effective insecticides now acquired by some insect species threatens to undermine much of the progress of the last decade in the chemical control of insect pests. The 'labelled pool' technique lends itself well to the determination, if any, of some fundamental difference in biochemistry between resistant and susceptible insects, by facilitating a quantitative comparison between certain biochemical processes in the corresponding tissues of the resistant and the normal insect.

Unique Techniques for Study of Insecticidal Action

The biochemistry section, it is recorded, now has at its disposal some unique and powerful techniques for the study of insecticidal action. Some promising lines of fundamental research have emerged, it is reported, which may ultimately prove to be of considerable practical value. These relate to the problems of synergism, selective action and resistance, and it is hoped to concentrate increasingly on these lines.

Insecticides and food: The study on the penetration of gamma-BHC into cheese has now been completed. After a period of 44 weeks from initial application, 40 per cent of the initial dose was found to be still present in the cheeses. The larger part of the residue remained in the outer few millimetres of the cheese. Residues exceeding 2.5 p.p.m. (recommended maximum concentration of gamma-BHC to be permitted in foodstuffs) extended only to a depth of 0.4 to 0.6 cm. below the surface of the Cheddar cheese and of 0.9 cm. below the surface of the Stilton. It has been concluded, therefore, that this use of gamma-BHC presents little toxic hazard to man. Repeated applications of this insecticide to Stilton cheese are inadvisable, however, because of the high residue built up.

Several chlorinated benzene derivatives have been detected in baked dough made from flour which had been treated with

gamma-BHC. The compounds have been identified in the form of their nitration products, which were separated by reversed-phase paper chromatography on a castor oil-impregnated strip with 60 per cent methanol as solvent. Further evidence of identity was obtained by hydrolysis of these chloro-nitro-compounds and separation of the resulting phenols by paper chromatography.

Although little information is available concerning the toxicity to man of the decomposition products, the report states that it seems unlikely that they would be more toxic than gamma-BMC. A detailed account of these studies is to be published.

Action of organophosphorus compounds: Studies have been continued on the mode of action of this 'increasingly important class of insecticides.' It has been shown that tetraethyl pyrophosphate (TEPP), as well as DFP, reduces the rate of acetylcholine synthesis *in vivo*. Reduction of rate of synthesis is thought to be an indirect effect of anticholinesterase action in the intact insect. Malathion, like DFP, was found to cause an accumulation of free glutamine in the insect thorax at the prostate stage, and also to stimulate oxygen consumption. These results are considered to suggest biochemical disturbances in addition to that of anticholinesterase action.

Resistance of house flies to dieldrin and to gamma benzene hexachloride is also considered in the report.

Swedish Designed Nitroglycerine Plant For ICI's Ardeer Factory

AFTER close on 40 years of production Imperial Chemical Industries' No. 1 Hill nitroglycerine batch plant at the Ardeer factory is being replaced by a new plant of Swedish design known as NAB. The new plant is expected to come into operation later this year. Work on the demolition of No. 1 Hill and preparation of the site for the new plant has begun. Construction is planned to allow No. 1 Hill to be kept in working condition up to the last possible minute. This, Mr. T. J. Tobin (nitroglycerine superintendent, blasting department) states, will enable delay in changeover to be cut to the minimum. Production will also be maintained.

The new plant has been chosen as the result of a visit made by Mr. Tobin and Dr. R. S. Gow, research department, to Sweden to see the new process. A pilot plant in operation in Sweden indicated definite advantages over any existing process of making nitroglycerine.

In the Swedish process, glycerine and nitric acid are reacted in an injector. The resulting emulsion of nitroglycerine and spent acid is passed to a centrifuge, where the nitroglycerine is separated from the spent acid. The process is a much safer one, since, in the processing, nitrating and separating, nitroglycerine in process is only about three per cent of the amount in any of the existing processes.

High-speed centrifuges, to be operated by remote control, will be housed in a reinforced concrete building, moulded over with earth works in the traditional manner.

Reorganisation at National Chemical And Warren Springs Laboratories

COUNCIL for Scientific and Industrial Research announces a change in organisation affecting two DSIR laboratories. Small steering committees are being set up to bear responsibility for the research programmes at Warren Spring Laboratory, Stevenage, and at the National Chemical Laboratory, Teddington.

The former research boards, which the steering committees replace, were responsible to the Research Council, but there was not necessarily any common membership. Under the new arrangement, the steering committees are full committees of the Research Council and the chairmen of the committees are members of the Council. In addition to the chairman there will be four members of each committee: two from industry, the secretary of the DSIR and the laboratory director. The committees are empowered to set-up specialist sub-committees as necessary.

The Warren Spring Laboratory Steering Committee is as follows: Sir Harry Jephcott *chairman* (chairman of the Research Council and Glaxo Laboratories Ltd.), Dr. R. Holroyd (deputy chairman, ICI Ltd.), Mr. D. A. Oliver, C.B.E. (director of research, BSA Group Research Centre), Sir Harry Melville (secretary DSIR), Mr. S. H. Clarke (director, Warren Spring Laboratories).

The National Chemical Laboratory Steering Committee, which will assume responsibility from 1 October 1958, will be as follows: Professor C. E. H. Bawn, C.B.E., *chairman* (member, Research Council), Dr. F. Roffey (research and personnel director, Distillers Co. Ltd.), Mr. B. Topley (director of development, Albright and Wilson), Sir Harry Melville, Dr. D. D. Pratt (director, NCL).

Petroleum Products Exports Up, Chemicals and Textiles Down

A review of UK exports during the months January to May 1958 shows that, apart from the engineering group, exports of only petroleum products revealed a marked upward trend, being increased in value by over £5 million. Chemicals, textiles, metals and manufactures in general were lower. The fall in textile exports this year reflects an almost general long-term world trend. Textiles fell from 16 per cent of the world exports of manufactures in 1951 to 10 per cent in 1956; their share in UK exports fell from 21 to 11 per cent.

Further Drop in U.K. Sulphuric Acid Consumption

SULPHURIC ACID AND OLEUM
Tons of 100 per cent H_2SO_4
1 April to 30 June

	Chamber and tower only	Contact only	Chamber, tower and contact
Stock, 1 April 1958	24,735	77,910	102,645
Production	118,424	413,495	531,919
Receipts	9,891	32,508	42,399
Oleum feed	—	764	764
Adjustments	−424	+247	−177
Use	75,018	246,802	321,820
Despatches	51,241	203,454	254,695
Stock, 30 June 1958	26,367	74,668	101,035
Total capacity represented	160,170	541,090	701,260
Percentage production	73.9	76.4	75.9

CONSUMPTION
1 April to 30 June 1958

Trade Uses	Tons 100% H_2SO_4	Percentage of total
Accumulators	2,964	0.55
Agricultural purposes	350	0.07
Bichromate and chromic acid	4,655	0.87
Bromine	2,464	0.46
Clays (Fuller's Earth, etc.)	2,291	0.43
Copper pickling	508	0.09
Dealers	2,715	0.50
Drugs and fine chemicals	4,801	0.89
Dyestuffs and intermediates	18,878	3.50
Explosives	2,670	0.50
Export	581	0.11
Glue, gelatine and size	151	0.03
Hydrochloric acid	12,897	2.39
Hydrofluoric acid	2,618	0.49
Iron pickling (incl. tin plate)	27,847	5.17
Leather	1,158	0.21
Lithopone	3,188	0.59
Metal extraction	560	0.10
Oil refining and petroleum products	12,370	2.30
Oils (vegetable)	2,670	0.50
Paper, etc.	1,543	0.29
Phosphates (industrial)	56	0.01
Plastics, not otherwise classified	10,492	1.95
Rayon and transparent paper	61,727	11.46
Sewage	2,495	0.46
Soap, glycerine and detergents	28,926	5.37
Sugar refining	128	0.02
Sulphate of ammonia	70,990	13.18
Sulphates of copper, nickel, etc.	3,308	0.61
Sulphate of magnesium	27	0.01
Superphosphates	117,921	21.89
Tar and benzole	7,985	1.48
Textile uses	2,595	0.48
Titanium dioxide	74,487	13.83
Unclassified	49,633	9.21
Total	538,649	100.00

Note.—These summaries exclude all Government plants.

RAW MATERIALS
Tons

	Pyrites	Spent Oxide	Sulphur: Imported	Sulphur: Recovered, H_2S and filter cake	Zinc concentrates	Anhydrite
Stock, 1 April 1958	217,942	108,824	65,387	10,710	135,330	9,245
Receipts	58,005	61,076	63,506	7,082	15,578	182,465
Adjustments	+278	+124	−206	+367	+309	—
Use	81,175	63,680	63,669	9,063	36,560	176,593
Despatches *	1,125	3,102	929	184	—	—
Stock, 30 June 1958	193,925	103,242	64,089	8,912	114,657	15,117

*Including uses for purposes other than sulphuric acid manufacture.

HIGH POLYMER CONFERENCE—3

Natta Considers Preparation and Properties of Graft Copolymers

REACTIVITY of the hydrogen atom linked to tertiary carbon atoms was greater compared to the hydrogen of a CH_3 or CH_2 group. While this reactivity could be considered inconvenient with regard to ageing stability at high temperatures of unstabilised polymers, it could be utilised to modify the properties of poly-α-olefins, particularly the surface properties and to prepare a very large series of grafted or reticulated derivatives. Shortly after the first preparation of isotactic polypropylene and polybutene, G. Natta (Instituto Chimica Industriale del Politecnico, Milan, Italy) began to study the thermal and catalytic peroxidation of such polymers, taking advantage of experience previously acquired in studying the low temperature peroxidation of cumene.

Monomolecular dissociation of the hydroperoxide did not take place or occur reversibly at low temperatures in the absence of free medical-capturing substances. Grafting, it was stated, took place rapidly, however, in the presence of monomers which could be polymerised by free radicals, even at relatively low temperatures.

In filaments under examination, which contained almost 85 per cent of crystalliable isotactic polymer (insoluble in boiling heptane), hydroperoxidation took place chiefly on the amorphous part. Almost one-half of the fixed oxygen was linked to the atactic polymers present in the sample in small amounts (5 to 6 per cent).

Rapid Peroxidation

Peroxidation, it was stated, could be achieved in a more homogenous way if it was carried out in solution in the presence of mutations. A suitable rapid peroxidation method of α-olefin polymers on a laboratory scale consisted in operating in cumene solution (and also in presence of small quantities of preformed cumene hydroperoxide). Side reactions were inhibited by 3 to 5 per cent of methanol. By bubbling air at atmospheric pressure into a 15 to 20 per cent solution of the polymer, peroxidation occurred rapidly at 65° to 70°C Cumene hydroperoxide and the poly-α-olefin hydroperoxides acted mutually as catalysts.

In the case of polymers such as partially crystalline polybutene which were easily swollen or soaked by initiator solutions, a rather homogeneous hydroperoxidation could be achieved with air. Hydroperoxidised polymers could be used as initiators for peroxidation reactions and could supply a variety of products by grafting, stated Natta. Surface hydroperoxidation of manufactured articles from isotactic polymers made it possible to modify the surface properties by grafting to produce, for example, hydrophilic fibre or films, dyeable fibres, etc. A concentration of 0.1 to 0.02 g. oxygen/m². of surface was, generally, sufficient to obtain a thickness of grafted polymers on the surface, which might vary from 0.01 to 0.1 mm.

Moderate grafting, unless followed by heat treatment, Natta reported, did not greatly modify the ultimate tensile strength and elongation of a filament. The high linearity of α-olefin atactic polymers and copolymers made them suitable for the production of very interesting grafted polymers. These could be prepared by dissolving the hydroperoxide of poly-α-olefin containing 0.2—0.4 per cent of peroxide oxygen in the monomer, and to operate under normal polymerisation conditions.

It was found that in the presence of unperoxidised poly-α-olefin, styrene did not polymerise at 80 to 90°C if no initiator was present. Addition of conventional initiators caused mainly homopolymers of styrene to form. In the presence of peroxidised poly-α-olefins (10 to 12 weight per cent) and under nitrogen at 80° to 90°C grafted polystyrenes possessing high impact strengths were formed. Those obtained by copolymerisation with or grafting of styrene on unsaturated rubbers showed, in general, a low stability ageing; but polystyrenes grafted on α-olefins showed mechanical properties which did not vary with ageing.

Grafting of vinyl chloride on poly-α-olefin proved difficult. The high solubility of amorphous poly-α-olefins in aliphatic hydrocarbons allowed non-grafted poly-α-olefins to be separated with ease from the homopolymers and from the grafted polymer of vinyl chloride. Poly-α-olefins did not show any affinity with p.v.c. and would separate out if they were not grafted.

Natta reported that the high number of tertiary carbon atoms clearly differentiated the poly-α-olefins from polythene, particularly when highly crystalline, and made the poly-α-olefin more sensitive to chemical reticulation and grafting processes which were milder and more easily controlled than high energy radiation processes.

Grafts of Polycaprolactam on Vinyl Copolymers

DESPITE complicating factors such as crosslinking reactions or incompatibility of the vinyl copolymer and polycaprolactam (nylon 6) preventing the formation of true graft copolymers, C. B. Chapman and L. Valentine (Department of Textile Industries, The University, Leeds 2) have shown that true graft copolymers were formed under certain conditions. The method of polymerisation used was essentially that of Flory and Schaefgen (*J. Am. Chem. Soc.*, 1948, **70**, 2709; US Pat. No. 2,524,046-1950).

It was considered that the reaction between vinyl polymers bearing reactive functions (e.g., COOMe, COOH, NH_2) and caprolactam should lead to the formation of grafted side chains of nylon 6 on a backbone of vinyl polymer. With styrene-methyl acrylate (S/MA) copolymers in particular, the reaction initiated by water at 220-250°C had been followed by measuring the melt viscosity, and the resultant polymers were characterised. Combination took place, but complications limited the extent of reaction, e.g., loss of amino groups, crosslinking, inhomogeneity of the reaction melt.

Soluble graft copolymers were formed only if the degree of polymerisation of the vinyl polymer was low, and the methyl acrylate content was high, e.g., a 1 : 1 S/MA copolymer of D.P.60 and 2 : 1 copolymer of D.P.150 both gave homogeneous graft copolymers. All other 1 : 1 and 2 : 1 copolymers of higher D.P. gave insoluble products, because of crosslinking.

For all copolymers in which the S/MA ratio was >2 : 1, very little reaction occurred, and the products could be separated by solvent extraction into three fractions, viz. nylon 6, unchanged S/MA copolymer, and a graft copolymer. At high S/MA ratios, irrespective of the degree of polymerisation, the two polymers were incompatible, and reaction was confined to the polyamide-copolymer interfaces. The soluble graft copolymers were characterised by elemental and end group analysis, melting points, melt viscosity, soluble viscosity, and infra-red spectroscopy.

From viscosity and fusion behaviour, it is believed that the soluble polymers obtained from the low degree of polymerisation polymers are highly complex. Crosslinking obviously involved the vinyl copolymer; pure polycaprolactam heated alone remained completely soluble, reaching a molecular weight of 60,000. Concentration of the polymer in the gel phase has also been observed.

Soluble products could be obtained, it was reported, by virtually complete hydrolysis of the gelled polymers, and hence it is suggested that it was unlikely that carbon-carbon bonds formed the crosslinks. The possibility of a reaction occurring between the ester groups and chain amide groups to form tertiary

amide crosslinks was put forward. A free-radical mechanism of degradation was indicated by the effect of the presence of α-naphthylamine. It was also noted that degradation was reduced in the presence of caprolactam monomer or polymer, indicating 'some further type of interaction with the vinyl copolymer'. Whereas oxidative degradation of polyamides led to darkening and loss of amino groups, no corresponding loss was observed with polycaprolactam. The plasticiser, apart from reducing melt viscosity, was found to have no appreciable effect on the compatibility of the polymers. Interaction of polycaprolactam with styrene/acrylic acid and styrene/maleic anhydride copolymers was clearly greater than for the acrylate copolymers, but crosslinking appeared to be more pronounced.

The main conclusion of Chapman and Valentine was that although graft copolymers could be made by polymerising caprolactam in the presence of vinyl polymers, bearing reactive groups, careful control was necessary to obtain pure products. Crosslinking reactions could be eliminated by using milder reaction conditions and an ester interchange catalyst with the styrene-methacrylate system. Modification of copolymer to provide greater compatibility with polycaprolactam would also favour formation of graft polymers, it was stated.

Irradiation Grafting Styrene to Polythene Films

COBALT-60 gamma irradiation of high pressure and low pressure (Marlex-50) polythene films immersed in styrene monomer to produce graft polymers was studied as a function of dose, temperature, and film thickness by Allan S. Hoffman (Massachusetts Institute of Technology, Cambridge, Massachusetts US). It was found that grafting generally increased with dose, but reached a level for all films at 10° to 20°C and an average dose rate of 228,000 rad/hr., where the grafting was higher for the thinnest films (1.5 to 2 ml.) than the thicker films (up to 20 ml.). At 40° and 70°C and an average dose rate of 93,500 rad/hr., the grafting increased approximately linearly with dose for all films.

Post-effects were also studied and found to be more significant for higher film thickness, higher polymer crystallinities, and lower temperatures. Observations of the heterogeneity found in the grafted films were described. This varied from a microscopic grating appearance on the surface to many bubbles under the surface or one large bubble along the length of the film. These bubbles contained a styrene-polystyrene solution. Linear films were less susceptible to bubble formation during irradiation at 10° to 20°C than at higher temperatures. The reverse trend appeared to be true for the branched films, although the bubbles were generally smaller than those for the linear films. The largest bubbles or pockets were observed on the thicker film at 40°C. It was concluded that for the dose rates employed, grafting was probably diffusion-controlled for all films at 10° to 20°C and perhaps for thicker films at 40°C. The effects of changes in the various dependent and independent variables on the grafting mechanism were discussed and a qualitative picture of this mechanism was described. It was then concluded that the different polycrystalline natures and different thicknesses of the films as they effected the monomer diffusion rate, and the dose rate or rate of initiation of free radicals were among the most important factors in determining the mechanism and resultant efficiency of grafting.

Synthesis of Graft Copolymers

PRIMARY purpose of the paper by G. Smets, A. Poot, M. Mullier and J. P. Bex (Laboratory of Macromolecular Chemistry, University of Louvain, Belgium) was to present some kinetic results obtained with polymethyl acrylate copolymers containing *tert*-butyl perester groups, in the initiation of the polymerisation of styrene. Further, on the basis of the kinetic scheme of decomposition of *tert*-butyl hydroperoxide in the presence of a copper salt, a new method of synthesis of block copolymers was presented which was based on the decomposition of *tert*-butyl peroxidic groups.

The two-step synthesis of graft copolymers was carried out by several methods. In the first method, the initial peroxidised polymer was prepared from a copolymer of methyl acrylate and acroyl chloride by reaction with *tert*-butyl hydroperoxide; the content of perester could be varied. The rate of decomposition of these perester groups and the degradation velocity of this first-step polymer were determined in benzene solution at various temperatures. The activation energies were 30.1 and 30.4 kcal./mole, respectively, which values correspond to the scission of the O—O linkage. Grafting experiments were carried out in dilute solutions (1 per cent) in order to avoid any grafting by transfer to the polymer.

The second-step polymerisation followed the square root law dependence with respect to the peroxidic polymer concentration. This rate was also of the first order with respect to the monomer, i.e., styrene, concentration. The rate of grafting was also evaluated by quantitative fractionation of the reaction products (precipitation method), taking into account the weight per cent of styrene in these isolated graft copolymers.

Rate of grafting was also of the first order with respect to the monomer concentration. A comparison of the total rate, R_p, and the rate of grafting, R_g, indicated that, at low perester-content, R_g was about one-half of R_p, while at high perester content, R_g was much lower than R_p as a result of the recombination of radicals. These kinetic results were discussed on the basis of a general reaction scheme.

In the second method, first-step polymers carrying *tert*-butyl peroxidic end-groups, were prepared on the basis of the decomposition of *tert*-butyl hydroperoxide in the presence of cupric octoate. The influence of the concentration of this copper salt on the rate of polymerisation has been considered. The first-step polymers so produced can initiate the polymerisation of a second monomer, so that block copolymers are obtained by chain extension, probably on both sides. In a third method, the initial peroxidic polymer was obtained by electrolysis of an aqueous solution of polymethacrylic acid. The experimental conditions were discussed. It was stated that the hydroperoxide-containing polymers, prepared and isolated at below 10°C, could be used for a second-step polymerisation with another monomer, e.g., acrylamide. Graft copolymers were thus obtained; these hydroperoxidic polymers were found to be relatively unstable, particularly because of the presence of neighbouring acid groups.

Ultra-violet Induced Crosslinking and Graphing of Solid High Polymers

PHOTOCHEMICAL aspects of ultra-violet-induced crosslinking and graphing of solid high polymers, particularly polythene, were described in detail by Gerald Aster, Gisela K. Osten and Harold Moroson (Polytechnic Institute of Brooklyn, New York). They reported that the production of crosslinks and of sites for grafting in solid high polymers could be achieved by hydrogen abstraction. This hydrogen abstraction could be carried out by means of far ultraviolet light (200-300 mμ).

Rate of the reaction was tremendously increased, it was stated, if certain sensitisers were incorporated into the plastic. A comparative study of various aromatic carbonyl compounds had been made to determine which were effective sensitisers. Crosslinking was accompanied by a photolysis of the sensitiser. Sensitisers which were effective for crosslinking were also effective for grafting. For the same degree of crosslinking the high density polythenes have greater strength at 150°C than have the low density polythenes.

The ultraviolet method differed from the ionising radiation method in that the dosages required for crosslinking by the former method were the same regardless of the degree of crystallinity of the polythene. A further difference was that whereas with the ionising radiation method extensive degradation occurred with certain polymers, in the ultraviolet

method crosslinking was the predominant reaction.

Details of cost of an ultraviolet lamp against a powerful ionising radiation installation was considered. In these workers' studies, a 15 watt GE germicidal lamp was employed which cost $5, but when it was desired to reduce the time of treatment a 1.2 kw type LL Hanovia lamp was used (cost $50). Using the type LL lamp, it is calculated that 50 sq. ft. of polythene film can be crosslinked in one minute.

Oster *et al.* said that the ultraviolet method resulted in an overwhelming crosslinking reaction, and even such polymers as polyisobutylene and polymethyl methacrylate, which were only degraded by ionising radiation, were readily crosslinked by far ultraviolet light. Polypropylene was also crosslinked by ultraviolet light. However, the ultraviolet method could not treat thick samples. The practical limit of penetration in some clear polythene samples equalled that of one-million-volt electrons (about 0.3cm.). Penetration was poor if the material scattered or absorbed light. Nevertheless, surface crosslinking and grafting could be effected even with carbon black filled polyethylene.

Graft Copolymer Formation During Polymerisation of Vinyl Acetate

AS it was believed that graft copolymers of polyvinyl acetate and polyvinyl alcohol could be formed in a polymerisation system using a water-soluble persulphate catalyst, methods of detecting, isolating and identifying them have been investigated by F. D. Hartley (Imperial Chemical Industries Ltd., Dyestuffs Division, Hexagon House, Blackley, Manchester 9).

Aqueous dispersions of polyvinyl acetate were prepared by polymerising vinyl acetate monomer, emulsified in water in the presence of polyvinyl alcohol and an alkali, with a water-soluble persulphate catalyst, polyvinyl acetate-polyvinyl alcohol graft copolymers being formed. The presence of graft copolymers in the dispersions was first established by the techniques of turbidimetric titration and adsorption and partition chromatography on paper. Subsequently the graft copolymers were separated according to species and at the same time the polyvinyl acetate was fractionated on a molecular weight basis, by using a continuous gradient elution chromatographic fractionation technique. The results obtained showed that in the fractionation of polymers by chromatographic methods, solubility in the developing solvent was a more important differentiating property than adsorption on the supporting medium.

Three dispersions prepared using identical quantities of vinyl acetate monomer and catalyst, but with varying quantities of polyvinyl alcohol, have been fractionated. Two different species of graft copolymer, one water-soluble and of high polyvinyl alcohol content, and the other water-insoluble and of low polyvinyl alcohol content, have been isolated from each of the dispersions. The relative amounts of the two species of graft copolymer and the molecular weight distributions of the polyvinyl acetate were found to differ in the three dispersions.

Mechanisms for the reactions giving rise to graft copolymer formation have been postulated which explain these differences. Thus it was suggested that the water-soluble species of graft copolymer were formed in the initial stages of the reaction by the radicals from the decomposing catalyst which first abstracted hydrogen atoms from the polyvinyl alcohol molecules to form polymer radicals which then initiates polymerisation of vinyl acetate monomer. Water-insoluble species of graft copolymer were formed at later stages in the reaction by transfer from growing polyvinyl acetate chains on to polyvinyl alcohol molecules, forming polymer radicals which can again initiate polymerisation of vinyl acetate.

In the infra-red spectra of both species of graft copolymer an absorption maximum occurred at a wavelength of 8.8μ. An absorption maximum occurring at this wavelength was characteristic of a tertiary alcohol group, and suggested that the grafted polyvinyl acetate chains were attached to the chain carbon atoms of the polyvinyl alcohol molecules to which the hydroxyl groups were also attached.

New Terephthalic Acid Process by Henkel and Cie

PRELIMINARY German patent DAS 1,027,656 by Henkel and Cie. GmbH, Dusseldorf-Holthausen, gives details of a new method of producing aromatic polycarboxylic acids from nitriles or amides. It permits several carboxylic groups to be introduced into any aromatic cyclic or heterocyclic molecule, even if other substituents are present and if the derivative is stable above 250°C.

A metal catalyst (zinc, cadmium, mercury, iron, lead or their derivatives) assists carboxylation which is carried out at pressures of 5 to 500 atmospheres. The product obtained is a salt of the polycarboxylic acid. For preference potassium carbonate is the reactant employed since this supplies alkali as well as the carbon dioxide required.

Products produced using this process are: Terephthalic acid from benzonitrile; 2,6-naphthalene-dicarboxylic acid from α or β—naphthoic nitrile; $\beta\gamma$—pyridine —carboxylic acid from β-cyanopyridine. Yields obtained are claimed to be of the order of 60 to 70 per cent.

B.P's Sulphur Recovery Plant at Isle of Grain Refinery

THE NEW sulphur recovery plant of British Petroleum Co. Ltd, at the company's Isle of Grain, Kent, Refinery, is reported to be currently working at about 60 per cent of rated capacity (*Sulphur* 1952, No. 21 (June)). The unit, which was designed by Simon-Carves Ltd., has a capacity of 45 tons a day high purity elemental sulphur. The sulphur recovery plant is based on a modified Claus kiln with two-stage conversion and after-burning equipment. Steam generated in the main waste heat boiler is used in the refinery, while that produced in the gas coolers is used for steam jacketing.

When in full operation the plant will be supplied with hydrogen sulphide containing gases from the refinery's catalytic cracker and the two hydrorefineries (designed by Kellogg International). Only one hydrorefiner has so far been completed under the major expansion scheme in hand for this refinery, but the second hydrorefiner is expected to be in operation during autumn this year.

Of the sulphur produced, some will be used to produce make-up acid in the Simon-Carves acid recovery plant. This plant will be fed with spent acid from the sulphuric acid alkylation plant and will produce 35 tons a day of 98 per cent acid. The rest of the sulphur may be disposed of either in flake form produced in Mitchell drum flakes or as molten sulphur dispatched in bulk road tankers. Initially, it is estimated that half the total of sulphur produced will be available for the domestic market.

Nuclear Power Station Site in Snowdonia National Park

The £50 million nuclear power station planned for Trawsfynydd, Merionethshire, is to be built for the Central Electricity Generating Board. The Board stated that the site is, with one exception, the only spot in the area which meets the exacting requirements for a nuclear power station. It is also hoped to build a larger station on another site.

The UK Atomic Energy Authority has announced that there should not be any danger from discharges into the lake.

1957 Figures for Industrial Injuries

The Ministry of Pensions and National Insurance reports that, under the Industrial Injuries Scheme, the number of claims for injury benefit in 1957 was 768,000, compared with 822,000 the previous year. Benefit costs for the year were £12¾ million. Industrial death benefit cost was estimated at £1,865,000.

ICI Pay Dispute for Arbitration?

Unions representing 55,000 workers employed by ICI Ltd. have rejected an offer of pay rises of up to 2d an hour (7s 4d a week). It is reported that unions representing the process workers intend to ask the Minister of Labour to refer the dispute to arbitration.

★ AN EASY way to cut costs has been discovered by the US Government, which has found that it is much cheaper to allow other people to do some of its work. A scheme which allows travelling scientists from the Army Chemical Corps to gather information from companies about their new compounds has 'gone into high gear' recently. The enthusiasm being shown is understandable, since estimates suggest that tests of each selected compound are costing the Government $50 (including travel and salary expenses) under the present arrangement. If the Army were to do all the preliminary work the cost would be as high as $1,000. Industry, too, benefits, for they hear of the results of the military laboratories' research.

At the moment the Chemical Corps' Industrial Liaison Office has only three scientists available to visit laboratories. Each man logs some 40,000 miles a year and visits about 120 companies, but the ILO would like to contact several thousand annually. Following visits paid last year the centre received about 2,500 ideas or compounds, and quite a number appeared to have possible military applications.

★ TESTS carried out by the Defence Standards Laboratories in Melbourne, Australia, have shown that some fabrics containing anatase can become unserviceable in a few months. It has been found that the extremely clear and bright Australian summer sun will cause fabrics made from rayon, nylon and Terylene to lose strength quite rapidly.

Dull and semi-dull fabrics are particularly susceptible, since the most commonly used dulling agent is anatase, a form of titanum oxide also used in paint manufacture, which accelerates the action of light on the fibre.

★ PIONEERS of new thinking in work study, productivity, welfare, safety campaigns and a host of other problems, ICI are literally blazing a new trail to the Continent. A co-ordinating committee is now being formed by the company to facilitate development of 'roll-on, roll-off' road transport to Europe.

This new service, which would be of great practical interest in a free trade area, was first started by ICI Dyestuffs Division, which recently began a regular service to the Continent, the first consignment reaching Tilbury on 1 June. Experiments were first held early last year when a 14-ton load was taken from Blackley to Rouen via Tilbury and Antwerp. The division now plans to send trailers on each of the three weekly Transport Ferry Service sailings (Mondays, Wednesdays and Fridays) destined for Germany, Italy, Yugoslavia, France, Belgium and Holland.

Charges levied by the Port of London simply to allow a trailer to be run on a ship at Tilbury are extremely heavy and constitute a tax on exports. This disadvantage should disappear as ICI extend the scale of operations; there would for instance be large savings because stocks held at various points on the Continent could be cut back.

ICI see no reason why the ferry service should not eventually be operated from other ports—Harwich, Dover or Hull, with the advantage of more realistic port dues.

★ VELBEX p.v.c. vinyl sheeting, made by BX Plastics, was chosen for the three-colour covers of a new set of attractive reference handbooks published by the Chemical Division of the Distillers Co. for their Bisol range of organic chemicals. This p.v.c. was selected because it is not affected by water nor by most chemicals.

The material was an appropriate choice since all the stages of its production from coal, limestone and salt —the basic raw materials—are carried out by DCL companies or their associates. DCL Bisol calcium carbide, with chlorine from Murgatroyd's Salt and Chemical, are used by British Geon to make Geon p.v.c. moulding materials. Geon p.v.c. and Bisol plasticisers are then incorporated by BX Plastics in Velbex sheeting.

This set of four books deals with Bisol solvents; plasticisers and other plastics chemicals; intermediates and miscellaneous chemicals; and Bisol methods of testing. Each contains a series of short monographs for each product.

★ 'TRADE effluents can contain almost anything (indeed I might have left out the qualifying 'almost'), but fortunately no one of them contains everything'; Dr. A. Key, chairman of the Ministry of Housing and Local Government committee on methods of analysis for sewage and sewage effluents, was speaking from rueful experience at a luncheon recently. His audience knew only too well what he meant, for at the time he was a guest at a luncheon in honour of the Association of British Chemical Manufacturers/Society for Analytical Chemistry joint committee which had just published 'Recommended Methods for the Analysis of Trade Effluents.' Dr. Key said that he did not think the invitation was meant to placate a rival (the Ministry's own 'Chemical Analysis as Applied to Sewage and Sewage Effluents' had been published previously) because he did not regard the two books as competing with one another. Methods of analysis best applicable to sewage were not always the most suitable for trade effluents, and vice versa.

Sometimes the two books, however, do cover the same ground. Oxygen demand tests are reproduced from the Ministry methods in the new recommendations with only slight modification, and all the recommended methods in the committee's booklet are 'in line' with Ministry policy.

Dr. Key ended his speech with a plea for more analysis of trade effluents. He stated that, if small rivers were to be maintained in a fit condition for all their proper uses and also dispose of increasing quantities of ever more complex liquid wastes, it was essential that we should learn what those wastes really were. He hoped the booklet would become well-known and widely used. Impatient analysers, however, who are not members of the ABCM or SAC, must wait until 5 September before they can obtain copies (through booksellers or W. Heffer and Sons Ltd., Cambridge, price 42s).

★ THE END of import licensing of dollar chemicals, announced by the Board of Trade last week to be put into operation on 18 August, comes as no surprise to the chemical industry. It was known that Government policy aims at the liberalisation of imports as soon as the dollar balance permits, in order to emphasise the position of sterling as a world-wide currency.

As far as chemicals are concerned, it is common knowledge that applications for import licences for essential materials, as distinct from luxuries, have been dealt with generously of late. It is, however, significant that the de-restrictions now announced apply to industrial requirements only and not to consumer goods.

★ A 'SERIOUS bottleneck' in silkworm production may be overcome as a result of some Japanese experiments. Scientists from the University of Kyushu have discovered that two chemicals found in mulberries seem to be the reason why silkworms find the leaves of these trees particularly attractive. Obviously silkworms change their tastes as they grow older—young larvae appear to be most stimulated by an alcohol, beta-gamma-hexenol, while older insects prefer alpha-beta-hexenol. Both chemicals should not be difficult to synthesise, if it should prove worth while 'doping' leaves more easily grown than mulberry ones to provide acceptable food for the insects.

Alembic

INDIA'S MAJOR DETERGENT PLANT IN OPERATION

Production Capacity 1,000 lb. per hour

IN THE Indian chemical and allied industries the most interesting news recently has been provided by new ventures on the part of laboratories and individual firms. A modern continuous plant set up by Swastik Oil Mills Ltd. for manufacture of the alkyl aryl sulphonate type of synthetic detergents went into operation recently in Bombay. The plant is the first major unit of its kind established in the country and has a production capacity of 1,000 lb. per hour. Tetrapropylene benzene required for the production of alkyl benzene is imported.

Ammonia recovery process. Ammonia recovery from coke oven gas by the Tata-Collins process is being introduced in the TISCO coke plant at Jamshedpur. The gas from the coke ovens will be treated with waste pickle liquor after it has been used in the sheet mills for galvanising steel sheets. The use of this waste liquor—which is highly noxious and corrosive—does away with the difficult problem of its disposal. The new process will yield, besides ammonium sulphate, other products such as hydrogen sulphide, ferrous hydroxide and cyanide sludge. The technique, patented under the name Tata-Collins process, was developed at the instance of the Tata Steel Co. by F. J. Collins and Co., Dortmund, W. Germany, and is being tried out for the first time on a commercial scale. Cost of this plant is approximately £540,000.

NCL Research

Synthesis of Vitamin C. Vitamin C has been synthesized at the National Chemical Laboratory, Poona, using D-sorbitol as the starting material. The process, carried out in four stages, is based on the original Reichstein method which has been modified to improve the conversion yields. The D-sorbitol used has to be imported. Pilot plant investigations on the preparation of sorbitol from glucose, which is indigenously available, are in progress. India's annual requirements of vitamin C are estimated at 75,000 lb.

Fusel oil titanate in insulating varnishes. Other investigations carried out at the laboratory have shown that fusel oil titanate (prepared from fusel oil, a by-product of the alcohol industry, by direct esterification with titanium tetrachloride) forms a good substitute for butyl titanate, which is now widely used to modify and improve the insulating and water-resistant properties of surface coating compositions. Varnishes prepared from fusel oil titanate silicate and cashew shell liquid formaldehyde resin have been found to possess superior electrical strength to the conventional varnishes.

Chlorinated turpentine. A new insecticide has been developed at the Regional Research Laboratory, Hyderabad, from chlorine and turpentine, both abundantly available in India. The process consists in chlorinating turpentine oil in a reaction vessel at low temperature by bubbling chlorine gas through the oil.

INDIAN NEWSLETTER

- **Alkyl aryl sulphonate detergent plant**
- **Tata-Collins ammonia recovery process**
- **Vitamin C and insulating varnish research at National Chemical Laboratory**
- **Raw materials production for paints**
- **Fertiliser plant at Neyveli**

Gas absorption is rapid and a viscous product containing about 68 per cent chlorine is obtained. A pilot unit set up at the laboratory is working satisfactorily.

Chlorinated turpentine is insoluble in water but soluble in common, organic solvents. It can be easily emulsified with water. Insecticides can be prepared in the form of solution in kerosene oil or water dispersible pastes and powders.

Tests against mosquito larvae (25 mg./sq. ft. of water surface) produced 100 per cent mortality after 24 hours. The insecticide, in various forms, has also been tested with some success as a contact poison against stored grain pests and as a kerosene oil composition for killing cockroaches, bugs, lice, termites, etc.

The director, Malaria Institute of India, has said that chlorinated turpentine compares favourably as an insecticide with DDT when tested under similar conditions.

Paint industry progress. The progress of the paint industry was reviewed at the general meeting of the Indian Paint Manufacturers' Association held recently in Calcutta. A significant advance has been recorded in paints and other surface coatings production in recent years. The industry is gradually building up an export market: the value of paints and varnishes exported has risen from £180,000 in 1954-55 to £262,500 in 1956-57. Production of the 50 units in the organised sector of the industry has risen from 27,950 tons (value £4.1 million) in 1950, to nearly 42,000 tons (value £9.75 million) in 1957. The Second Five-Year Plan has fixed a target of 60,000 tons for paints and varnishes to be achieved by the end of 1960-61.

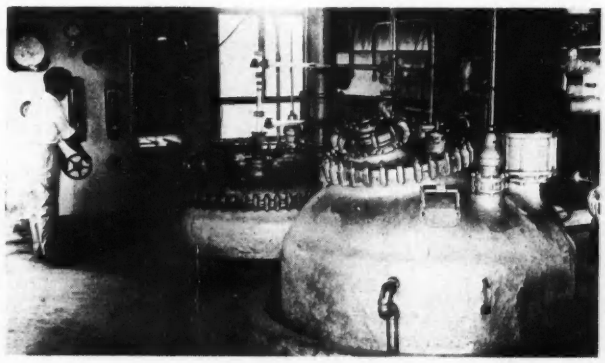
Raw Material Schemes

In the raw materials field production of alkyd resins, anatase titanium dioxide and aluminium paste pigment has been established. New raw materials schemes under implementation or consideration are: Pigments—rutile titanium dioxide, phthalocyanine blue, prussian blue from iron oxide sludge; synthetic resins—phenolic resins pure and modified, maleic resins; plasticisers—dibutylphthalate; solvents—butyl and amyl alcohols, butyl and amyl acetates, glycol ethers and esters, methyl ethyl ketone, methyl isobutyl ketone, diacetone alcohol; basic chemicals—phthalic anhydride, pentaerythritol, urea, phenol, and formaldehyde. The developments will greatly facilitate production of synthetic and nitrocellulose finishes.

Present imports of raw materials are valued at £11.25 million.

Neyveli lignite project. The installation of a pilot briquetting and carbonising plant is the first stage of the £56.25 million project now being worked by the Neyveli Lignite Corporation on a five-year scheme to exploit the lignite resources at Neyveli for a period of about 60 years, quarrying about 3.5 million tons a year. A fertiliser plant to be erected there will use up 0.5 million

(*Continued in page 226*)



Alcohol chlorinator at DDT factory, Alwaye—one of the new projects mentioned in our issue of 19 July

NEW KNAPSACK PROCESS FOR ACRYLONITRILE

MANUFACTURE of acrylonitrile, by a process which uses acetaldehyde and hydrocyanic acid as the raw materials, is announced by Dr. K. Sennewald and Karl-Heinz Steil of Knapsack-Griesheim AG, Knapsack, near Cologne (*Chemie-Ingenieur Technik*, 1958 (July) p. 446). Following successful laboratory trials on the process, a 30 tons a month pilot plant was set up and has been tested for its technical possibilities in all respects.

Features of the new process are:

(1) A 10-15 per cent higher yield than in the process for making acrylonitrile from acetylene and prussic acid (tantamount to a corresponding reduction of raw material costs.

(2) A further economy of raw material costs (about 10 per cent) is achieved by using cheap acetaldehyde from an ethylene basis. (Under these conditions, 20 to 25 per cent of the raw material costs can be saved. As raw materials account for about two-thirds in value of the cost price of acrylonitrile, acrylonitrile produced by the Knapsack process is claimed to be 13 to 14 per cent cheaper than the product obtained by the acetylene-prussic acid process).

(3) Acrylonitrile produced by the new process is of high purity and eminently suitable for the production of high grade emulsions and block polymerisates (confirmed by technical use of the product on a 10-ton scale).

(4) Production of acrylonitrile is no longer tied to the location of acrylonitrile production and can be carried out in any locality since the starting materials for the new process, such as ethyl alcohol, acetaldehyde or lactic acid nitrile, can be transported.

(5) The new process has the advantage that stocks of hazardous hydrocyanic acid are no longer required (lactic acid nitrile can be stored without any risk).

When plans were made by Knapsack for a major acrylonitrile production by the acetylene process, the moderate yield and impurity of the acrylonitrile obtained were carefully considered. It was known that acetaldehyde, cheaply available from industrial-scale production, formed with hydrocyanic acid a cyanhydrin, α-oxypropionic acid nitrile, CH_3-CH(OH)-CN or lactic acid nitrile. Unlike β-oxypropionic acid nitrile (ethylene cyanhydrin), produced by reacting ethylene oxide and prussic acid, lactic acid nitrile is highly unstable and at higher temperatures is easily split into acetaldehyde and hydrocyanic acid. Experiments were therefore undertaken to convert lactic acid nitrile into acrylonitrile.

It was assumed that dehydration of lactic acid nitrile could be accomplished chiefly by quick heating to the dehydration temperature and also by selection of suitable catalysts. Lactic acid nitrile, however, showed a tendency to decompose into the starting components, acetaldehyde and hydrocyanic acid.

A. Wolfram and his associates at Knapsack eventually succeeded in the following way: Lactic acid nitrile was mixed with 80 per cent phosphoric acid and the mixture was introduced in the form of a very finely divided dust into a vacuum furnace, in which it was rapidly (fraction of a second) heated to 600°C and, after being held for a short time at this temperature was cooled by injection of water down to room temperature. In this way it was possible to heat about 50 per cent of the lactic acid nitrile, with decomposition to the reaction temperature, and dehydrate it to acrylonitrile. The remainder of the lactic acid nitrile, apart from small amounts decomposed in other ways, broke up into the starting materials. acetaldehyde and hydrocyanic acid.

In order to avoid vacuum operations, heating of the finely divided reaction mixture under normal pressure with hot, oxygen-free combustion gases of about 1,500°C to a reaction temperature of about 600° to 700°C was investigated. It was found that the proportion of lactic acid nitrile converted into acrylonitrile was increased to 67 to 70 per cent.

In considering the development of the process on an industrial scale, recovery of the acetaldehyde and hydrocyanic acid by recombination of these two decomposition products to lactic acid nitrile is of importance. By re-using the recovered nitrile, it was found possible to increase the acrylonitrile yield to over 90 per cent of the lactic acid nitrile originally used. Phosphoric acid used in the reaction, after recovery (in a concentration of 30 per cent) from the reaction gases, was re-used in a continuous cycle. It was very simply reconcentrated to 80 to 85 per cent.

Technical details of the Knapsack process revealed show that acetaldehyde and hydrocyanic acid react easily to form lactic acid nitrile. Speed at which the lactic acid nitrile forms is stated to depend on the pH value (pH value of 7 is sufficient on the industrial scale). The nitrile can be produced either in batches or continuously. To produce lactic acid nitrile, hydrocyanic acid is introduced into a reactor vessel supplied with arrangements for efficient cooling and powerful agitation. Sufficient 20 per cent caustic soda is added to bring the pH of the solution to 7 to 7.5. The exactly equivalent amount of acetaldehyde is then added with cooling and agitation. On completion of the reaction the lactic acid nitrile formed is stabilised by addition of 0.2 to 0.5 per cent phosphoric acid. The material obtained is of 97 to 98 per cent purity and can be used directly for acrylonitrile production. Using the batch method on the present Knapsack semi-scale plant 100 tons a month are being produced. For a larger plant a continuous process could be readily designed, it is reported.

Essentially the operations carried out using the Knapsack process are:

(a) Splitting lactic acid nitrile into acrylonitrile and water in the presence of phosphoric acid by direct action of hot combustion gases at about 600°C; about two-thirds of the lactic acid nitrile is converted into acrylonitrile while one-third breaks up into hydrocyanic acid and acetaldehyde;

(b) the hot gas stream is cooled and the phosphoric acid in 30 per cent concentration is separated by circulating 30 per cent phosphoric acid; the recovered phosphoric acid is reconcentrated to 85 per cent;

(c) Lactic acid nitrile is recovered from the cold gas stream by recombination of the hydrocyanic acid and acetaldehyde contained in it;

(d) Acrylonitrile is obtained from the gas stream in the form of an aqueous solution by scrubbing with water. Further processing to give crude acrylonitrile corresponds to that in the acetylene-hydrocyanic acid process.

Use of phosphoric acid at such high temperatures, with regard to corrosion as well as deposition of condensed phosphoric acids on the timing of the reaction furnace, had to be considered. From experience in operating the plant, it was found that corrosion problems could be completely overcome by use of appropriate materials or suitable *modus operandi*. By suitable design it proved possible to prevent completely deposition of condensed phosphoric acid leaving the reaction furnace. It was noted in particular that the phosphoric acid leaving the reaction furnace (recovered as a furnace condensate in a 30-35 per cent concentration) did not contain any significant amounts of pyro-, meta- and poly-acids.

Total acrylonitrile yield at present amounts to 90 to 92 per cent of the amounts of acetaldehyde and hydrocyanic acid used in the process.

The product obtained by this new Knapsack process is from the outset free of unsaturated impurities, which themselves tend to polymerise, or which may encourage acrylonitrile to polymerise or may impair the quality of the polyacrylonitrile produced. That the product is highly suitable for making high grade emulsions and block polymerisates, has been confirmed by technical use of the product on a 10-ton scale.

Indian Newsletter

(*Continued from page* 225)

tons of lignite to produce 152,000 tons of urea, with a fixed nitrogen content of 70,000 tons per annum, at a cost of £15.75 million. Specifications of the plant have been sanctioned by the Government subject to foreign exchange credit facilities being available.

Overseas News

U.S. REDUCED STEEL PRODUCTION AFFECTS COAL CHEMICALS OUTPUT

COAL chemicals production in the US is expected to follow the level of steel output closely for the rest of the summer and it is anticipated that it will be at a relatively low level for the next couple of months, rising in the autumn with the expected seasonal pick-up in steel plant activity (*Chem. and Eng. News*, 1958, **36**, No. 29, 23).

A delicate balance is reported to exist among the big-volume coal chemicals, particularly with regard to benzene, toluene and xylene, which can all be produced by petroleum refineries.

For the steel industry, coal chemicals are by-products. Today, steel output in the US is stated to be running at a little over 50 per cent of capacity and coke oven production at just over 60 per cent. The average steel production for the first six months of this year is reported to be only 54 per cent, while coke ovens are reported to have operated at 62 per cent capacity. This is 35 per cent below the same period last year for furnace plants, more than 25 per cent lower for merchant plants, which account for about 15 per cent of total coke production in the US.

The US car industry represents a big market for steel and also uses a fair amount of products derived from coal chemicals.

Affecting US coal chemicals are imports, such as benzene from Iron Curtain countries (available at 25 to 30 cents per gallon, a price well below domestic quotations, it is reported). In 1957 imports made up about 15 per cent of US sales. In mid-July last, US benzene prices were dropped by about 5 cents a gallon to about 31 cents, because of the petroleum refiners' intention to retain and gain markets. Sales of imported benzene for this year as a whole are reported likely to be down, even to half those of last year. Also, whereas 1 million lb. of naphthalene were imported in 1957, and Russian stocks are available now at 3 or 4 cents a lb., domestic material is quoted at 7 cents.

With steel requirements being reduced, it is likely that coke-oven rates will slow down thus changing the balance of production of coal chemicals. With dropped operating temperatures more toluene, xylene and creosote is produced in the product mix and less benzene and naphthalene. However, even with shifts in production anticipated for certain coal chemicals, supplies are not expected to become short. Also, prices of coal chemicals are not expected to rise in view of competition from refineries and imports.

Fertilisers Plant In Egypt

A Franco-German Group (UHDE, Badische-Anilin, and Schneider) has secured a contract to build a plant for the production of fertilisers in Egypt. The new plant will cost an equivalent of £22 million and its maximum daily capacity will reach 1,280 tons. A similar plant near Suez(produces only 200,000 tons a year.

Agreement on S. African Uranium Oxide Production

Following discussions between British and US experts and the S. African Atomic Energy Board, it has been agreed that, from July 1958, no more than 6,200 tons a year of uranium oxide should be sold by the Union to the Combined Development Agency. Last year's total uranium oxide output by S. African producers was 5,709 tons, all of which was bought by the agency. Any uranium produced in excess of 6,200 tons may be sold elsewhere than through the agency, and the contract terms between the board and the agency remain unchanged.

Acetic Acid Plant for India

The Hyderabad Construction Co. Ltd., is reported to have obtained a licence from the Central Government to build an acetic acid plant in Hyderabad with a capacity of 6 tons a day. Machinery for the plant is to be supplied on a deferred-payment basis by A. B. Chematur of Stockholm.

French Plutonium Production

At Marcoule, Southern France, a new atomic reactor went into operation on 21 July and marks France's entry into large-scale production of plutonium. By the end of the year, the plant will be producing plutonium at an annual rate of 40 kilos and feeding more than 30,000 kw. of electricity into the national grid.

A smaller atomic reactor is already in operation and a third is expected to 'go critical' by the end of the year, bringing France's plutonium output to 100 kilos a year by 1959.

Portugal to Produce More Fertilisers

To provide for a substantial increase in the home consumption of nitrogenous fertilisers and an exportable surplus, Portugal is planning to spend escudos 890 millions (about £11 million) on a production expansion programme.

The Sociedade Portuguese de Petroquimica and Nitratos de Portugal are being equipped to produce from refinery by-products a supply of town gas, 80,000 tons of nitrogenous fertiliser and 12,000 tons of ammonia a year for the ammonium sulphate industry. Sapec is to manufacture 70 tons of nitrogenous fertiliser a day.

Steel works coke ovens are to produce 60,000 tons of ammonia a year, bringing the total additional output planned from 1961 onwards to between 75 and 100 thousand tons.

In 1956, Portugal's home consumption of fertiliser nitrogen was 52,718 tons, or 15.4 kilogrammes per hectare. The target now aimed at is a minimum of 90,000 tons, equivalent to 26.1 kilogrammes per hectare, in 1964.

Chemicals in Australian Free-imports List

Announcing that Australia would maintain the import licensing level for the next four months at the present annual ceiling of £A800 million, Mr. J. McEwen, Trade Minister, listed goods to be exempt from import licensing as including annato piezo electric quartz plates, bars and rods, bromine salts, various cyanides and hydrosulphites, resin, cobalt, magnesium vanadium and arsenic metals, chrome silicide, calcium silicide, molybdenum and chromium metal.

Firestone's New Synthetic Rubber

Firestone Tire and Rubber Co. US, have announced a new synthetic rubber. Known as Diene, the product is produced by polymerising butadiene with a lithium-derived catalyst. Diene is described not as a complete rubber in itself, but as a natural rubber extender. It is understood that the company is producing Diene in the same pilot plant as is used for Coral brand synthetic natural rubber.

Exact chemical structure of Diene has not been released as yet. However, the new product is stated to be substantially different from butadiene polymers obtained with Ziegler-type catalysts. A 'substantial portion' of Diene in truck tires is stated to improve crack resistance and skid resistance of tyres.

Austrian Chemical Exports Rise By 20 per cent.

Gross output of Austria's chemical industry in 1957 was £107 million, about 13 per cent more than in 1956. Exports rose 20.3 per cent to £26.5 million. The value of medicinal and pharmaceutical preparations produced in Austria reached £5.1 million, of which about 13.2 per cent was exported. Sales of magnesite and magnesite products now run at more than £14 million a year, output being 1,172,600 tons in 1957. Austria's magnesite deposits are among the largest in the world.

Investments in Czechoslovakia's Chemical Industry

A marked increase in investments in Czechoslovakia's chemical industry is not expected this year. The funds put at the disposal of this sector for new plants will exceed those for 1957 by 21 per cent. (In 1955 the amount exceeded by 78 per cent that of 1954.)

growth of investments will be on a par with the increase in the concentration of investments. More than 77 per cent of the whole of the investments will be concentrated in 13 enterprises, of which 3 alone represent 31.3 per cent of the whole. This year it is hoped to construct a sulphuric acid factory, and another factory for the production of nitrogen fertilisers, ammonia and paper.

Montecatini's Activities In Iran

Societa' Montecatini have signed an agreement with the Development and Resources Corporation regarding the erection in Iran of a petrochemical plant which will produce p.v.c. and other plastics materials.

More Chemicals Imported By Dominican Republic

According to the 'Exporter's Guide to the Dominican Republic', that country's imports of chemicals and pharmaceuticals are increasing rapidly. Figures for the last three years are as follows: $4.9m. (1955); $6.2m. (1956); $7.6m. (1957). Sections of the guide deal with the market, trade statistics, import regulations, business procedure, communications and 'six golden rules' for the exporter.

Quebec Firm to Double Size of Ligno-Sulphonate Plant

Lignosol Chemicals Ltd., Canada, are to double the size of their Quebec lignosulphonates plant. Sales of the company are reported to be up 25 per cent from 1957.

Ammonia Synthesis And 'Air-Gas' Process

Work by Dipl. Chem. H. H. Kopper and Prof. Dr. E. Wiske, Hamburg (reported in *Chemie-Ingenieur-Technik* 1958, **30**, 545) has shown that the mass-transfer resistance between the gas current and the (external) catalyst surface in ammonia synthesis has practically no influence on the course of the chemical reaction. In carbon-dioxide gasification of coke ('air-gas' process) this resistance is stated to be only of significance in the conversion of materials under special conditions. The results obtained make it appear impossible to transfer to these heterogeneous chemical reactions the methods developed on the basis of heat and mass transfer for phase transition, as was recently attempted.

Shell Refinery Not For S. Africa?

The Shell Co. of South Africa Ltd., recently announced that it hoped to establish a £25 million oil refinery on 300 acres of reclaimed land in the Salisbury Island area of Durban Bay. Planned capacity of the plant would be in the vicinity of 4 million tons a year, or some 1,000 million gallons of petrol and fuel oils. The Durban City Council is, however, at present considering a recommendation from its general purposes committee that the company be refused permission to establish a refinery there because of possible air and water pollution. If the plant had to be built further away from Durban harbour than now planned, it has been suggested that the Shell group might find it more economical to erect the proposed refinery in another country altogether.

Consumption Of Petrochemicals In Italy

During 1957, Italy consumed 90,100 tons of petrochemical products in manufacture of artificial and synthetic textile fibres. This total exceeds by 7.1 per cent the total recorded in 1956, as can be seen from the detailed table given below:

	1956	1957
	Tons	
Phenol	9,600	12,200
Paraxylol	170	400
Ammonia	2,000	2,500
Methanol	100	200
Ethylene glycol	100	200
Carbon disulphide	45,000	42,000
Acetic anhydride	12,500	14,500
Acetone	1,500	1,500
Total	84,670	90,100

Spanish Plant To Increase Polystyrene Production

The northern Spanish chemical company, Union Quimica del Norte de España, SA, Uniquinesa, are reported as intending to increase polystyrene production at their works at Bilbao from 1,200 to 3,000 tons annually. Cost of the project will be 40,825,000 pesetas including 23,950,000 pesetas for the import of machinery and plant mainly from West Germany. Raw materials worth 39 million pesetas are to be imported per year.

New Polyolefin Resin Series

US Celanese Corporation have introduced a new polyolefin resin-family—Fortiflex B. These are described as linear polyolefins with a few short branches along the main chain. The product is stated to show improvement in resistance to loading and stress, without losing any of the desired properties of linear polyolefins.

First commercial product of these new polyolefin resins is designed for monofilaments, but other products are expected to be in production this month.

No details have been released as to composition or method of preparation of the new resin. It appears to be a copolymer based mainly on ethylene. The other component, it is suggested, may be butylene.

Alcohol Producing Licences Granted In India

Licences for the establishment of new alcohol manufacturing undertakings have been granted to the following companies or departments by the Indian Government: Commerce and Industry Dept., Government of Madhya Pradesh, Bhopal, for a new plant at Ratlam, M.P.; Cudh Sugar Mills Ltd. for an extension to their plant at Hargaon (Sitapur), U.P.; M/s Walchandnagar Industries, Bombay, for plant at Poona; Government Alcohol Factory (Agents Hyderabad Construction Co. Ltd.), Hyderabad, Dn., for plant at Shakarnagar Distt. Nizamabad; Karnal Distillery Co. Ltd., Karnal, for plant at Rathdama Distt. Rohtak. Capacities of the new plants range from 30,000 to 100,000 gallons per month of power and industrial alcohols, etc.

Chilean Nitrate Output Up 13 per cent in 1957

Two companies have been granted new nitrate concessions in the province of Tarapaca, Chile; the Cia. Salitrera de Tarapaca y Antofagasta is to exploit two concessions spread over 134,000 hectares, with an estimated content of 1.2 million tons, and Empressa Luis de Urruticoechea are to work the Pampa Mayo deposit of 33,000 hectares containing about 88,000 tons.

Total production of Chilean nitrates in 1957 was 1,309,534 tons, compared with 1,159,176 tons in 1956. Copper production was slightly down at 450,631 tons, and iron ore much the same at 2,679,663 tons.

USSR Petroleum Reforming Research

Use of a platinum on silica gel catalyst for reforming a naphtha fraction has been the subject of a study by chemical engineers working at the Division of Chemical Sciences of the USSR Academy of Science (*Izv. Akad. Nauk USSR*, 1957, **10**, 1223, reported in *Platinum Markets Review* 1958, **2**; No. 3, 91).

Preparation of the catalyst was by impregnation of silica gel with a solution of chlorplatinic acid (platinum concentration 0.5 per cent). Particle size range of the gel was 1.5 to 2.5 mm. The hydrocarbons in the naphtha fraction had a boiling range from 96 to 114°C and were, by volume, 55 per cent paraffinic, 39 per cent naphthenic and 6 per cent aromatic. The apparatus used was of conventional design with an electrically heated stainless steel reactor. It was arranged for gas-recirculation and was suitable for operation at pressures up to 50 atm.

The catalyst proved to be an effective dehydrogenating agent, the aromatics coming chiefly from dehydrogenation of naphthenes. The optimum combination of conditions for the maximum production of aromatic hydrocarbons was established. The effects of pressure, hydrogen/hydrocarbon ratio, space velocity and temperature on the yield and composition of the product were studied. Altering the gas ratio, it is stated, had little effect; increasing the pressure and the space velocity diminished the yield of aromatics. Raising the temperature increased the aromatic hydrocarbon content.

EQUIPMENT REVIEW

Chemical Plant: Laboratory Apparatus Safety and Anti-Corrosion Products

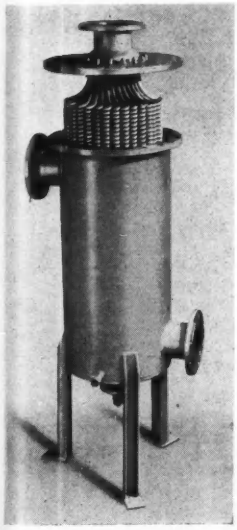
ALLOY GATE VALVES

An improvement, for which patents are now pending, has been made to the design of alloy gate valves manufactured by Langley Alloys Ltd, Langley, Slough, Bucks. Specially machined faces on the wedge or gate of the valve, consisting of a series of concentric annular bands with grooves between are employed in place of the normal plane faces. This feature, known as the 'Multi-seal' is now available on all Langley Alloy Gate Valves of sizes 1½ in. bore and larger in the 150, 300 and 600 classes.

The company state it has been proved by many tests that the life of the seating faces is more than doubled before re-seating is necessary even when the valve is made in austenitic stainless steels and the wedge and body seat facings are of the same alloy quality. Without the use of plastic, rubber or other non-metallic seatings, the shut-off is yet said to be complete and positive, even when body movement takes place due to temperature variations, etc.

HELITUBE HEAT EXCHANGER

An 'unconventional' heat exchanger has been devised by British Boiler Accessories Ltd., 62-3 Fenchurch Street, London EC3. The design utilises a fabricated steel jacket casing enclosing a



B.B.A. Helitube heat exchanger

nest of copper, steel, aluminium or stainless steel tubes. The Helitube heat exchanger surfaces consist of narrow tubes tightly coiled into a helical shape, and these are secured in the tube plates by means of a tight fitting ferrule which beds the tube into grooves. If steel tubes are used they are welded into the tube plates. These plates are rigidly fixed in the tube jacket without the necessity for expansion plates or floating heads. Movements caused by variations in temperature are taken up in the springy action of the tubes.

High overall heat transfer is achieved, say the company, since the tubes are of a small bore, normally not more than ½ in. diameter, which ensures intimate contact with the heat transfer surfaces of all the liquid, and at the same time turbulence is maintained. The B.B.A. Helitube is manufactured at the works of the parent company, T. H. and J. Daniels Ltd., Stroud.

KEEGLAS REINFORCED PLASTICS

Presented by Kestner Evaporator and Engineering Co. Ltd. under the name of Keeglas is a new range of reinforced plastics. The company announces that the mechanical properties have proved to be such that reaction vessels may be supported upon Keeglas frames. A recent example is a cylindrical vessel for mercuric chloride with a capacity of 250 gallons or one ton. This is 4 ft. 3 in. diameter and 3 ft. 6 in. deep, and is also fitted with a Keeglas shaft and propeller. Because no reinforcing other than Keeglas is used, the supporting framework is inert to corrosive attack.

Two standard grades of Keeglas are now readily available. Both are suitable for working up to 100°C, but one can withstand a maximum working temperature of 130°C. Alternative grades are also available for special duties (e.g., a glass fibre and furane resin laminate backed by glass fibre and polyester resin laminate, as well as combinations of epoxy resins and polyester resins with glass fibre base in the form of a double laminate). Pipes, tanks and reaction vessels are also being fabricated by Kestner Evaporator and Engineering Co. Ltd, 5 Grosvenor Gardens, London SW1.

DIGITAL DISPLAY UNITS

Two firms are now producing in-line digital display units. The indicators are designed to display a selected number in clear, illuminated figures.

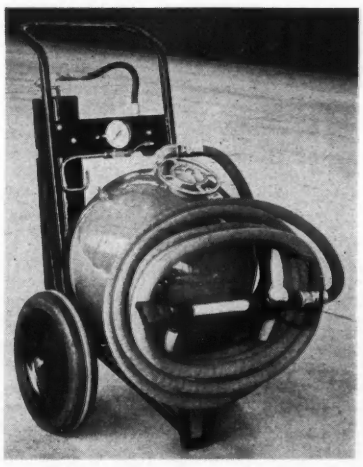
Hilger and Watts Ltd., 98 St. Pancras Way, London NW1, are manufacturing indicators in two types: edge-illuminated, and projection. The company provides a choice of white or green light and continuous or dotted numerals. In the edge-illuminated type, each unit (one decade) comprises ten Perspex plates each engraved with a numeral from 0 to 9. The order in which the plates are stacked can be varied, and the plates engraved with signs, etc., as required. The projection type are for situations in which a display needs to show from a distance or wide angle.

Counting Instruments Ltd., 5 Elstree Way, Boreham Wood, Herts, make a unit which displays a digit 1 in. by ⅝ in. The standard model from this company has provision for projecting the figures 0 to 9 and two decimal points. Other models are being developed by the firm showing letters, etc., and with provision for either coloured characters against a black background or a coloured outline to a white character. Price per unit is in the region of £6, and one suggested use for a bank of these units is as a read-out on a digital volt meter.

TEMPERATURE SCANNING EQUIPMENT

Fielden have developed from their potentiometric scanning systems an automatic temperature scanner to monitor up to 50 temperature points. The temperature is indicated on a large dial approximately 2 ft. diameter and around the periphery of this dial are the point numbers e.g. 1-50. Since the desired value for all points will not always be the same, it has been made possible to have a different set point for each of the 50 variables. The value of the set point is shown by the position of a red pointer. The actual value of the temperature is shown by the position of a green pointer and the measuring circuit is potentiometric.

The equipment has three tolerance bands which can be adjusted to required values. Any number of temperature points can be allocated to any of these three bands. This will then enable, say the makers, the most important variables to give an alarm should they deviate by more than, say, 2 per cent, others may

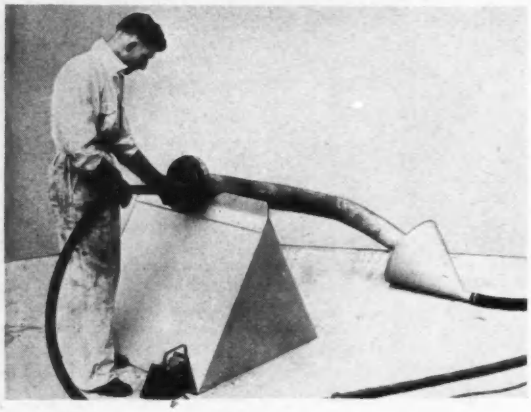


The 'Fireball' 150-lb. dry chemical extinguisher produced by Foamite Ltd., 235/241 Regent Street, London W1

be set to give an alarm should they deviate by more than, say, 5 per cent or perhaps 10 per cent. In normal operation the equipment will scan the 50 points in sequence, but manual over-ride is possible and this enables any temperature point to be indicated at will.

TUBE CLEANING EQUIPMENT

A claim that a small range of simple attachments enables the cleaning of practically any shape or size of tube from $\frac{1}{8}$ in. bore upwards is made by Vacu-Blast Ltd, Bath Road, Slough, Bucks. The machine itself incorporates a pressure/vacuum system for abrasive feed and recovery with a segregation of grit, slag and dust.



Vacu-Blast tube-cleaning equipment

With tubes of $\frac{1}{8}$ in. bore the blow-through method is used, but the cross-sectional area of the subject items must be greater than the area of the blast nozzle. Usually a nozzle of 5/16 in. bore is used and tubes of $\frac{1}{8}$ in. bore are nested to give a cross-sectional area upwards of 0.077 sq. in. An adaptor is made to nest these tubes. Tubes of bore sizes $\frac{1}{2}$ in. to 2 in. may also be cleaned on the blow-through principle, but the nozzle is now offered direct to the mouth of the tube. Above this size the nozzle can be entered into the tube and traversed right along the work surface.

Special attachments may be required for cleaning tubes of 3 in. bore and above. Supersonic or parallel bore nozzles are made, as is a spider attachment to centralise the nozzle in the tube.

Cleaning speed varies according to the nature and condition of the tube to be cleaned, and the equipment will not tackle pipes containing a thick oily or glutinous substance which absorbs the impact of the abrasive.

TWO 'TRIANGLE' VALVES

For the petroleum and chemical industries Triangle Valve Co. Ltd., Lamberhead Green, Wigan, have produced two new valves. Their line blind valve is designed to eliminate the need for special tools when closing the blind. It uses the wedge gate principle to give positive leak-proof shut-off. The seal is provided by the line pressure exerting itself against 'O' rings in the wedge-disc faces. Maximum service temperatures are 250°F with synthetic rubber or 450°F with 'Teflon' or 'Fluon'.

The company's cast alloy steel 'Wellhead' lubricated through-conduit gate valve has an inside screw and non-rising stem. Plastic gland packing is provided and the body chamber is filled with lubricant.

ELGASTAT EXPORT MODEL

Elgastat B.102/Ex portable deioniser has been specifically designed for use overseas and is fully tropicalised. The model produces high purity water at up to 17 litres/hour; flow commences instantly at a turn of the tap. The deionised effluent is at least equal, the company announces, to triple glass distilled water and is free from trace metals, silica, chlorides, sulphates, ammonia and carbon dioxide. An integral conductivity meter, provides constant quality check.

To simplify procedure for overseas buyers there is a standard price of £39 ($109.20 US dollars) for the B102/Ex. These prices include packing, insurance and freight to any part of the world. Producers: Elga Products Ltd., Railway Place, London SW19.

ROYLES TWIN STRAINERS

To improve the operating efficiency of their twin strainers type "G" Royles Ltd, Irlam, Manchester, have motorised the valve changeover gear and have fitted a flame proof electronic switch to control the motor, automatically switching off at the end of the valve travel in either direction. Additionally, hand wheels are provided for emergency manual operation.

In this new design the two strainer cages are not removable, but access is provided by quick release cleaning doors.

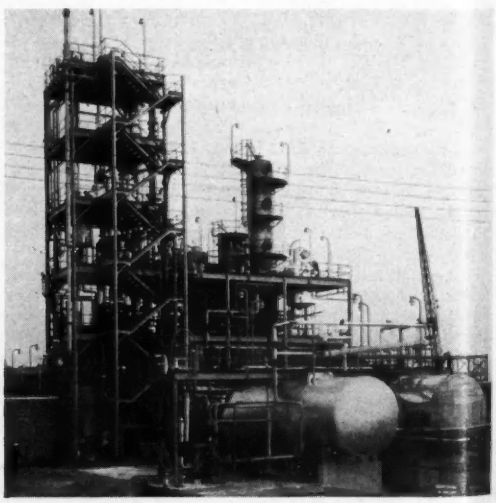
FIRE ALARM SYSTEM

The "Gent 1151" system has been developed to give complete automatic warning in any business or factory premises, and to the local fire brigade, of a dangerous rise in temperature. The system has received the approval of the Fire Offices' Committee.

The design uses a mercury tube to eliminate dirty or corroded contacts. Complete testing facilities have been incorporated: by pressing one test push at the indicator control panel the system can be tried out 'in a matter of seconds'. The test can be immediately acknowledged by the Fire Brigade on the equipment without the necessity of telephoning.

Gent and Co. Ltd, Faraday Works, Leicester, claim that the arrangement of the electrical circuit allows considerable installation-cost savings to be made.

Chemstrand's N. Ireland Acrilan Plant



The solvent manufacturing and recovery section of the Acrilan acrylic fibre plant now nearing completion at Coleraine, Co. Londonderry, Northern Ireland. The plant, which is being built for Chemstrand Ltd., will have an initial capacity of 10 million lb. of acrylic fibre per annum and should be in production before the end of this year

Chemist's Bookshelf

CATALYTIC REACTIONS

CATALYSTS. Vol. V. Hydrogenation, Oxo-synthesis, Hydrocracking, Hydrosulphurisation, Hydrogen isotope exchange and related catalytic reactions. Edited by *P. H. Emmett*. Reinhold, New York, and Chapman and Hall Ltd., London. 1957. Pp. vi + 542. 120s.

This volume in Professor Emmett's impressive series covers a fairly wide diversity of topics. The first three chapters are devoted to those catalytic reactions of carbon monoxide not covered in Volume IV. The remaining three chapters deal with various aspects of 'hydrogenation' in the comprehensive meaning of this word, i.e. including the addition of hydrogen accompanied by rupture of chemical bonds in the reactants for which the term hydrogenolysis is a convenient description. As Volume III and IV were also mainly based on the same theme of 'hydrogenation', it is natural that in this third and last volume on the subject there is a number of rather varied topics to be treated.

The chapter by M. Orchin and I. Wender on 'Reactions of Carbon Monoxide' covers a range of different kinds of reactions and illustrates the versatility of this compound for the syntheses of various kinds. The account is well documented and it is noteworthy that a high proportion of the work quoted refers to the patent literature. The same two authors are joined by H. W. Sternberg for the chapter on 'The Oxo Reaction'. This is a description generally used for the process whereby olefines are converted by the addition of hydrogen and carbon monoxide to aldehydes and then to a variety of secondary products. A good account is given of the various investigations which have been made on the mechanism of this reaction.

The selection of three authors from Italy, G. Natta, U. Colombo and I. Pasquon, for the chapter on the direct synthesis of higher alcohols from carbon monoxide and hydrogen is a happy one. Much of the work on modifications of the catalysts used in the synthesis of methanol in order to produce higher alcohols originated from that country, and this is the main topic of the chapter.

H. A. Smith has produced an admirable and comprehensive account of the work which has been carried out on the hydrogenation of aromatic compounds in the fourth chapter. He has arranged in a clear and readable presentation material drawn from over 400 references. In some ways it is surprising how little is yet known about the fundamental mechanisms involved in the addition of hydrogen to the aromatic nucleus.

The fifth chapter by T. I. Taylor on 'Hydrogen Isotopes in the Study of Hydrogenation and Exchange' is excellent. It occupies some 150 pages and covers not only results obtained in recent years, chiefly through the use of mass spectrometric techniques, but also the earlier work of the 1930's, among which that of A. and L. Farkas occupied such a prominent place.

The final chapter by J. B. McKinley on 'The Hydrodesulphurisation of Liquid Petroleum Fractions' reviews a field of great industrial importance. The successful elimination of unwanted sulphur compounds from crude petroleum has become a problem of growing importance as methods for the catalytic treatment of petroleum have developed. McKinley, like other authors in this volume, pays due attention to the fundamental thermodynamic considerations relating to the reactions he describes and gives some useful tables of such data.

The standard of presentation and the quality of the diagrams are high, and the comprehensive author index and subject index are valuable features. There can be little doubt that this volume maintains the high standard of the earlier volumes in the series and it can be recommended most strongly despite the high price.

C. KEMBALL.

Presenting Technical Material

ORAL COMMUNICATION OF TECHNICAL INFORMATION. By *R. S. Casey*. Reinhold, New York; Chapman and Hall, London, 1958. Pp. vi + 199. 36s.

Often after delivery of scientific papers, complaints are heard from the audience that the technical material has not been presented well. There are also complaints from the industry that graduate chemists and engineers cannot present their information clearly, either orally or in writing. Robert S. Casey, author of over 30 journal articles and various chapters in books and encyclopaedias, outlines the actual problems involved. The book starts, in effect, with Chapter II, Bridges and Barriers—which has the typical American psychological approach. Other chapters cover organisation of material, composition, delivery of formal and inpromptu speeches, and the use of mechanical aids such as recordings and slides. Also dealt with are: how to preside at meetings, talk science to laymen, give technical legal testimony and converse socially. Throughout the author has borne in mind the special needs of technical subject matter.

In the reviewer's opinion the most valuable chapter is that dealing with 'audio and visual aids'. In this country, graphs, diaphrams and other illustrative matter are usually poorly reproduced. Casey covers sound transmitting, sound recording and sound equipment; projection equipment, easel displays, standards and specifications for visual aids (e.g., notes on legibility which are particularly valuable), standards for comprehension and design of copy (maps, diagrams, photos). Details are also included for the construction of visual aids, such as graphs, simplified sketches, preparation of slides, and a quick note on models and exhibits. Secretaries of societies and other organisers may also find the chapter on 'Presiding at Meetings' helpful.

In all, a useful book for the large company's library shelf. It is felt that the price, however, bars this book from personal acquisition. D.L.M.

Current Australian Work on Heterocyclic Compounds

CURRENT TRENDS IN HETEROCYCLIC CHEMISTRY. Edited by *Albert*, *Badger* and *Shoppee*. Academic Press Inc., New York; Butterworths Scientific Publications, London. 1958. Pp. ix + 170. 30s.

Heterocyclic chemistry has too long been the Cinderella of organic chemistry, and it is gratifying to see a new publication on this important subject. This volume is a record of the proceedings of the 1957 Symposium, sponsored by The Chemical Society, and held at the Australian National University, Canberra. It comprises twenty short and unrelated papers of varying quality, describing current Australian interest in the chemistry of heterocycles. The title of the book is thus slightly misleading unless one understands 'in Australia' in parentheses. Viewed in this light, it is a praisworthy publication which merits the attention of teachers and research workers interested in this branch of chemistry. In subject matter the papers range over the chemistry of natural products, the synthesis and reactions of simpler molecules and the biological role of heterocyclic compounds. Not the least stimulating feature of the book is the inclusion of the discussion which followed each paper.

Some of the most interesting organic chemistry in the book is to be found in the four alkaloid papers. These describe work on furoquinoline, pyrrolizidine, quinolizidine and aporphine alkaloids, and with the exception of the last mentioned each is admirably illustrated by formulae. It is regrettable that the paper on 'Hernandine—a new aporphine alkaloid' contains no formulae whatsoever. On the same high standard is an account of the stereochemistry of the catechins and related

Chemist's Bookshelf

flavan derivatives. There are also interesting reviews of work on the synthesis of monosubstituted phenanthridines and of pyrimidine nucleosides, and on the chemistry of pteridines and of sulphur heterocycles. A few of the papers are so brief and deal with such narrow fields that they will have difficulty in arousing or sustaining the interest of the average reader.

The production of this book leaves nothing to be desired: the type is clear, the formulae and diagrams are clean and of appropriate size, and the text is free from all but trivial errors. Formulae V, VI and VII on p. 2 include imperfect cyclopropane rings, and the formulae on p. 126 have B_z misprinted as B_s. On p. 83 the second paragraph contains an error in orientation, and a misleading reference to formula III.

Any book of this type has a limited life, and the publishers have wisely produced it at a modest price.

G. L. BUCHANAN.

Surveys of 140 Industrial Chemicals

INDUSTRIAL CHEMICALS. 2nd Edition. By *W. L. Faith and Others*. Chapman and Hall, Ltd., London. Pp. ix + 844. Price 128s.

One hundred and forty industrial chemicals from acetaldehyde to zinc oxide are discussed in this second edition. The arrangement is similar to that used in the first edition which contained 106 chemical surveys (and 652 pages). Each chemical is treated individually in a separate chapter, supplying the following information on each: how it is made (with important process details), how it is used; how much has been produced each year for the past 20 years; the price range for the past 20 years; the important chemical and physical properties of the material; the usual grades; how it is packaged and shipped; who makes it and where; important economic trends. The only change in form of this edition is a listing of names and locations of manufacturing plants to replace showing the locations on maps as in the first edition. All production and price charts and use patterns have been brought up to date and the economic aspects of each chemical have largely been rewritten. It is regretted, however, that no references are included regarding the source of process data or statistical information.

Those familiar with the first edition will undoubtedly wish to have this second edition, particularly in view of the quite extensive revisions, such as additional information on phenol, including the cumene peroxidation process. Surveys of chemicals included for the first time are: acetanilide; alkyl aryl sulphonates; furfural, hydrazine, hydrogen cyanide, hydrogen peroxide, lithium carbonate, monosodium glutamate, titanium dioxide, 2,4-tolylene diisocyanate, vinyl acetate, vinyl chloride and zinc oxide.

D.L.M.

Third Volume of "Klages" Discusses Specialised Products

LEHRBUCH DER ORGANISCHEN CHEMIE. Vol. III: Band Sondergebiete. By *F. Klages*. Walter de Gruyter, Berlin. 1958. Pp. xix + 776. DM.98.

Several years have passed since the publication of the earlier volumes of this textbook, and 'Klages' now enjoys well-deserved popularity among undergraduates and research workers. Volume I, which appeared in two parts, deals with systematic organic chemistry, and Volume II provides an excellent survey of theoretical aspects. The present volume, which completes the set, is devoted to the detailed discussion of more specialised topics.

The first of the three opening chapters, all of which deal with industrial subjects, gives a brief account of chemical aspects of the formation and utilisation of organic 'minerals' like coal and oil. It is followed by a chapter on dyes, which embraces biological pigments as well as industrial products, and includes a useful discussion of the relation between colour and constitution. The third chapter summarises the fundamental physical and organic chemistry of macromolecules.

The remainder of the book is devoted to biological products and processes. It includes sections on carbohydrates, terpenes, steroids, alkaloids, proteins, enzymes, hormones, vitamins and antibiotics, as well as an excellent survey of biochemical reactions.

Considering the wide range of topics in this volume, it is perhaps inevitable that some of the sections should fall below the high general level of the whole textbook. Thus the chapter on carbohydrates contains many errors, and the reviewer was disappointed to find no mention of conformational analysis in the sections on terpenes and steroids. However, on the whole, Professor Klages has been most successful in spotlighting fundamentals, and his many excursions into borderline topics, such as the structure of wood and the biogenesis of alkaloids, make stimulating reading. The material is very well organised, and the chapter on biochemistry deserves special mention as a masterpiece of lucid condensation. The book contains much recent work; vitamin B_{12}, macrolide antibiotics and the synthesis of strychnine are all mentioned, and there is an excellent section on the mechanism of photosynthesis. Numerous literature references to key papers and recent review articles are a valuable feature.

PETER SCHWARZ.

Analytical and Chemistry Know-How

QUANTITATIVE CHEMICAL ANALYSIS, 11th Edition. By *L. F. Hamilton* and *S. G. Simpson*. The Macmillan Co., New York. 1958. Pp. xvi + 566. 41*s*.

In the new edition of this well-known American text-book the authors have attempted to condense a wide range of classical gravimetric and titrimetric procedures and a selected range of modern instrumental methods, with fairly full theoretical discussions of each topic, into an elementary text-book of reasonable size. The text throughout is strewn with helpful practical hints, worked-out problems and a full range of numerical and review questions. The general impression conveyed by this book is that it is absolutely crammed full with chemistry and analytical know-how. The authors have succeeded in producing a book that is of immense practical value, and one which is so diverse in its approach and wide in its coverage that it is difficult to envisage a college course where it could not be used successfully with little if any modification.

The book opens by classifying analytical chemistry into several divisions and then deals with general operations and basic physico-chemical principles. Part II, devoted to titrimetric analysis, treats the usual range of acid-base, redox and precipitation titrations and has a brief chapter on complex formation methods. Part III on gravimetry starts with a well balanced treatment of general principles and goes on to deal with a few selected methods and the analysis of an ore. Part IV on instrumental methods—physical methods would have been a more appropriate name—gives an account in simple terms of electro-deposition, potentiometry, conductimetry, amperometric and coulometric methods and briefly surveys the whole field of optical methods in the visible region including polarimetry and spectrography. The section ends with an outline of gas-analysis methods. Part V, on miscellaneous methods, gives working details for the analysis of minerals, ferrous and non-ferrous alloys, and various technical products such as foods, drinking water, cereals and bleaching powder.

It should perhaps be stressed that the book ceases to be a manual of working method in Part IV, but this does not constitute a serious criticism, for the book is intended only for first year students and this section has the merit of being well written and easy to assimilate. It is stimulating, too, to find that the authors take cognizance of solvent extraction, chromatography, ion-exchange, EDTA titration and some other modern non-instrumental methods, even though no experimental methods are given.

A very sound and attractively written introductory text for students in technical colleges and universities.

T. S. WEST.

Chemist's Bookshelf

Encyclopedia of Chemical Reactions Covers Newer Metals

ENCYCLOPEDIA OF CHEMICAL REACTIONS. Vol. VII. Compiled by *C. A. Jacobson*, edited by *C. A. Hampel*. Reinhold Publishing Corporation, New York; Chapman & Hall Ltd., London. 1958. Pp. 479. 102*s*.

The 'Encyclopedia of Chemical Reactions' is a well-known work to be found in most technical libraries. The first six volumes dealt with the inorganic reactions of elements from aluminium through to sodium and (Vols. 1-5) were published until the death of Professor Jacobson interrupted the work. The series was continued in 1956 under the editorship of C. A. Hampel, who now presents the seventh volume.

There are 1,753 entries in this book. These are distributed as follows:—strontium, 56; sulphur, 558; tantalum, 51; technetium, 23; tellurium, 215; terbium, 7; thallium, 227; thorium, 162; thulium, 6; tin, 314; titanium, 115. The indexing and recording of the reactions follows the familiar pattern and the book ends with a formula index to 'Reagents' and another to 'Substances obtained'.

The fundamental concept behind this massive publication is most praiseworthy and its execution in this new volume is in keeping with this ideal. Nevertheless the result of all this labour leaves one with a vague feeling of dissatisfaction. The entries are essentially in the nature of a skeleton card index—one must accept this as an unfortunate necessity in a work of this character—but even so, the 'index' appears to be very incomplete and sometimes obsolete. For example, reference to work such as Mellor (1924), Hopkin's "Chapters in the Chemistry of the Less Familiar Elements' (1942) and Sneed and Brasted, 'Comprehensive Inorganic Chemistry' Vol. IV (1955) bear out this contention in relation to thulium and terbium. For terbium, the most modern reference given in the 'Encyclopedia' is 1908; for thulium, 1911. Naturally much work has been done on these elements during the past 50 years.

However, the compilation of these abstracts represents a useful contribution to chemistry and the new volume will be welcomed by all who possess the earlier series. One must judge the price in relation to the specialist nature (and therefore limited sale) of the book, but nevertheless in my opinion, it is too high.

T. S. WEST.

Survey on Gypsum and Anhydrite

'GYPSUM and Anhydrite' is the title of an Overseas Geological Surveys monograph by the late Dr. A. W. Groves (HM Stationery Office, price 7s 6d). The monograph quotes statistics to show the very rapid increase between 1954 and 1956 of Britain's use of anhydrite for making sulphuric acid.

Imperial Chemical Industries Ltd. (Billingham Division), Solvay Chemicals Ltd. and United Sulphuric Acid Corporation by the end of 1955 all had large plants for the conversion of anhydrite in operation. In 1954 the ICI sulphuric acid plants were extended to produce around 180,000 tons of acid a year. Production at the Solvay plant in Whitehaven commenced in 1955 (annual capacity 100,000 tons), as did manufacturing at the Widnes plant of United Sulphuric Acid Corporation (potential consumption estimated at 240,000 tons of anhydrite per annum, from which about 148,000 tons of acid and 132,000 tons of cement clinker should be produced). The St. Bees Headland mine, which supplies the Solvay plant, is expected to produce 7,000 tons of anhydrite a week, about half of which will be sent to other works.

The expansion in UK consumption of this material was mainly due to the difficulty during 1954-6 of obtaining elementary sulphur from the US.

It has again become cheaper to manufacture acid from elementary sulphur than from anhydrite. 'Nevertheless,' it is stated, 'the increased use of anhydrite can be justified not only because it reduces dependence on imported acid-making materials but because it permits the production of cheaper acid than is possible when using pyrites, which at present accounts for the largest item in the country's sulphur import bill.' The prospect for the anhydrite industry is thought to be one of steady growth.

The UK production of gypsum plus anhydrite has increased from one million tons in 1938 and the early war years to 3.3 million tons in 1956. Following a review of gypsum production, the monograph concludes that regions where gypsum industries may in time be initiated or where, if suitable local deposits of gypsum cannot be located, a gypsum products import trade might be fostered, include much of Africa and such Far Eastern centres as Singapore and Hong Kong. Factors which have to be borne in mind, however, include the possibility of cheaper local competitive materials and the difficulty of handling calcined gypsum in moist tropical climates.

BA's New Address

The British Association for the Advancement of Science have now moved to 18 Adam Street, Adelphi, London WC2.

Dollar Import Licensing: Further Details

The new Open General Import Licence (No. 2) issued by the Board of Trade (see CHEMICAL AGE, 2 August, p. 194) comes into effect on 18 August. A 'Notice to Importers No. 866' from the Board's Import Licensing Branch, 43 Marsham Street, London SW1, gives further details about the coverage of the new licence. In almost every case it is specifically stated that goods packed for retail sale will not be de-restricted.

The following information is not complete and needs to be checked against the licence itself, but some of the more important items are shown below.

Materials covered by the licence Most separate chemically defined inorganic and organic chemicals. Fatty acids; acid oils from refining; fatty alcohols; ethyl alcohol; natural barium carbonate; kieselguhr; diatomite; lime; cryolite; natural arsenic sulphides. Oils and other products of the distillation of high temperature coal tars and similar oils and products obtained by other processes, and products of a like nature derived from petroleum. Petroleum gases and gaseous hydrocarbons. Many fertilisers (including potassium chloride and certain grades of calcium cyanamide; calcium nitrate; sodium nitrate; urea and ammonium phosphates). Tannins and synthetic tanning substances. Certain pigments; and many inorganic products used as luminophores; prepared pigments; opacifiers; prepared driers. Many essential oils and resinoids; organic surface active agents; albuminoidal substances and glues including casein adhesives, albumins, peptones and dextrins. Artificial graphite; sulphite lye; spirits of turpentine; certain artificial resins and plastics materials. Naphthenates; sulphonaphthenates; alkylbenzenes, mixed; alkylnaphthalenes, mixed; alkenes, mixed; polyoxyethylene glycols, liquid; fusel oil; catalysts consisting of a chemical product, or finely divided metal, on an inert support; certain hardening agents; certain antioxidants; certain metallic carbides; desiccating agents mainly of silica gel; alkaline iron oxide preparations used for coal gas purification; glycerol monostearate; glycerol distearate; urea-hydrogen peroxide.

Exceptions to the licence Radioactive chemical elements and radioactive isotopes; compounds, inorganic or organic, of such elements or isotopes, whether or not chemically defined. Salts and other compounds, inorganic or organic, of thorium, uranium or intermixtures of such salts and compounds. Calcium carbide; sulphonamides; provitamins and vitamins; hormones; antibiotics; pigments rendered radioactive; aqueous distillates or solutions of essential oils; lubrication preparations containing 70 per cent or more by weight of petroleum or shale oils; compound disinfectants, insecticides and similar products. Dyestuffs and dyestuff intermediates.

CHEMICAL PIONEERS

16 Ernest Solvay

Subject of the 16th in the series of chemical pioneers by Dr. D. W. F. Hardie is Ernest Solvay. Although Solvay's soda process was not a new chemical discovery, his chemical engineering techniques were so far in advance of anything else at the time that he was granted international patents.

ERNEST SOLVAY was born on 16 April 1838 in the Brabantine village of Rebecq-Rognon. His father, Alexandre Solvay, in addition to being a quarry-master, carried on a wholesale trade in oil and soap, and—significant for the career of his son—he was a small-scale salt refiner. When Ernest was 17 severe illness put an end to his hopes of attending university. He passed his long convalescence helping with his father's account-books and reading what works on chemistry and elementary physics were available to him. In 1859 he was sent to work with his uncle Semet, who managed a gasworks at Schaarbeck, a suburb of Brussels. About a year later he was made assistant manager, under his uncle, at another suburban gasworks in St. Josse-ten-Noode.

Ammonia Liquor Disposal

In 1860 the gas industry had not solved the problem of disposing economically of the dilute ammonia liquor it produced. The low concentration made it unprofitable to transport it. At the St. Josse gasworks Solvay successfully addressed himself to the business of producing strong ammonia. There was a limited outlet for ammonium carbonate at that time, and Solvay began experiments on its production from gasworks liquor, which contained various carbonates of ammonia. Solvay made a casual experiment to discover how salt behaved with ammonium carbonate in the presence of a small quantity of water. The result surprised Solvay—neither salt nor carbonate appeared to dissolve. He pursued the matter further and discovered that the undissolved crystals were not in fact the original salt and ammonium carbonate, but sodium bicarbonate, resulting from their interaction: $NH_4\ HCO_3 + NaCl \rightarrow NaHCO_3 + NH_4Cl$.

At this stage Solvay believed that he had discovered a new chemical reaction; his reading in applied chemistry had not then been wide enough to acquaint him with the already long history of failure of attempts to turn this reaction, originally discovered by Fresnel in 1811, to industrial utility as a simpler alternative to the LeBlanc process for the manufacture of soda. Solvay knew enough, however, of the importance of the alkali industry immediately to realise the great potentialities of his discovery.

In April 1861 Solvay was granted a Belgian patent for the use of the supposedly new reaction as the basis of a soda-making process. With his younger brother Alfred, an engineer, he set up a small plant near the Schaarbeck gasworks. All the combined enthusiasm and skill of the brothers was necessary to meet the difficulties and accidents that beset them, but by the middle of 1863 they had developed a workable process. The reason for their success on the technical side was that—although they did not yet know it—their chemical engineering had been superior to that of those chemists in Scotland, England, France, Germany and Austria who had sought the same goal.

If Solvay had not made a chemical discovery, he and his brother had invented what was then more important—a plant in which the reaction could be effectively applied.

It was while negotiating for financial support from a Belgian Minister of State, Primez, in order to exploit the process on an industrial scale, that Solvay became aware that he had been anticipated. This was no small shock to the young inventor. 'I quickly recovered,' he wrote, 'and was sustained by the conviction that in my apparatus for bicarbonation I had an advantage over my predecessors.'

Apparatus Patented

In international patents granted in 1863 (BP 3131/11 Dec. 1863) Solvay laid no claim to the reaction, but only to an apparatus 'by means of which the formation of carbonates of soda is rendered practically available for manufacturing purposes.' The principal feature of the plant protected by the patent was the carbonator in which the carbon dioxide, under pressure and dispersed in small bubbles, travelled a total distance of about 100 yards through the ammoniated brine. Indicative of Solvay's chemical engineering ingenuity is the utilisation of the difference in density between brine and ammoniated brine to effect automatic and continuous supply of the ammonia to the process. With the financial support of Primez and, ultimately, also the Solvay family resources, Solvay and his brother formed a company with a capital of 136,000 francs. The new factory was built at Couillet, near Charleroi. All technical difficulties had not been overcome, and five months after the factory came into operation the daily output of soda ash was only 500 lb.

It is not true, as is commonly supposed, that the economics of the new process gave it an instant advantage over the LeBlanc system. Operationally the 'wet' process, as it was then called, had the advantages that it was simpler, continuous, and worked at low temperature. Because of its continuous and low temperature operation the Solvay process had lower requirements of labour and fuel. In 1872 a ton of Solvay soda cost about £2 less to produce than a ton of LeBlanc soda. The LeBlanc manufacturer, however, was compensated for this disadvantage in soda-making costs by the then very large profits being made from bleaching powder, produced from the by-product hydrochloric acid from his saltcake furnaces. Even the most optimistic observers of the time saw ammonia-soda expansion limited to supplying demand for soda in excess of that co-produced with the amount of bleaching powder that could be sold by the LeBlanc manufacturers. It was also confidently predicted that, due to increased demand, the price of ammonia would rise—in fact it fell, because of its increased recovery from coke ovens. To this last development Solvay and his Semet relations made an important contribution in the form of the Semet-Solvay oven, which greatly increased recovery of ammonia.

By the early 1870's news of the great success of the process at Couillet had spread far beyond the frontiers of Belgium. Ludwig Mond wasted no time in visiting Couillet and, in 1873, having secured a two-year exclusive licence of Solvay's British patents, erected his ammonia-soda factory on the Cheshire saltfield. In the years immediately following, the Solvay process was introduced into France and Germany. The position, however, still remained that the Solvay process menaced only one flank of the LeBlanc system; the chlorine 'half' of the salt used in the ammonia-soda process went to waste in the calcium chloride effluent from its ammonia recovery stage. Until a new chlorine process made its appearance the LeBlanc system held its own in the war of processes. The awaited new chlorine process made its appearance in the 1890's in the form of various inventions for the manufacture of chlorine and caustic soda by the electrolysis of brine.

Solvay, as he observed the growth of the chemical industry, became more and more aware of the importance to it of the advance of pure science. Out of the profits of manufacture he made very large benefactions for the establishment of institutes for the study of chemistry, physics, physiology, and sociology. His mind had a philosophical cast: he saw all the sciences as a unity serving the social advance of mankind.

He was a man of great physical as well as mental energy. For many years he walked daily from his home outside Brussels to his office in the Rue Prince Albert. At the age of 65 he made the ascent of Mont Cervin, which he had scaled in earlier life, and three years later he was climbing in the Mont Blanc group of mountains. He died at Brussels on 26 May 1922.

Chemists' Federation Agrees Restrictive Trading Case

THE CHEMISTS' Federation and the Registrar of Restrictive Trading Agreements were congratulated by Mr. Justice Devlin, president of the Restrictive Practices Court in London, on the measure of agreement they had reached, and he hoped that it might be used as a precedent in other cases as the right course to be followed whenever possible.

Mr. J. R. Cumming-Bruce, for the Registrar, said that there was a high degree of confidence between the parties. Both were assured that no surprises would be sprung and that the salient features of their cases would be made known in detail by 16 September.

PEOPLE in the news

● After 21 years with the Beecham group of companies, MR. H. G. LAZELL has succeeded Lord Dovercourt as chairman of this £33 million enterprise.

Mr. Lazell was a director and secretary of Macleans Ltd., when that company was acquired by Beechams in 1938. He became secretary of the group the same year and a director of the main board and managing director of Macleans in 1940. He became managing director of the group in May 1951.

Three distinct achievements are attributed to Mr. Lazell. He streamlined a loosely-knit collection of companies handling dozens of different products in widely separated fields into five compact autonomous entities. He backed his belief in research as a means of keeping ahead of world competition by stepping up expenditure on this account from £50,000 in 1943 to £425,000 last year. Thirdly, under Mr. Lazell's leadership, Beecham's have had a dramatic success in North American markets by a policy of single-product concentration.

● DR. J. D. ARMIT has been appointed a director of Triplex Holdings Ltd. Dr. Armit, formerly a director of ICI and wartime director-general of explosives at the Ministry of Supply, has for some years been consultant chemist to the Triplex group.

● MR. G. P. BALFOUR, B.Sc., A.M.I. Chem.E., has relinquished his position as managing director of Sharples Centrifuges Ltd., Camberley, to become managing director of Stockdale Engineering Ltd., the constructional chemical engineers of Poynton, Cheshire, in succession to Mr. S. Hopton. Mr. Balfour has had a wide experience of oil refinery design and in recent years has worked on methods of liquid-solid and liquid-liquid separation.

Mr. G. P. Balfour

● MR. C. H. J. CORBETT and MR. T. WILLIAMS, directors of the Eaglescliffe Chemical Co. Ltd., Darlington, have been appointed managing directors of the firm.

● SIR WILFRED NEDEN, C.B., C.B.E., chief industrial commissioner of the Ministry of Labour is to retire on 24 August, to be succeeded by MR. P. H. ST. JOHN WILSON, under secretary of the industrial relations department since January 1957. Mr. Wilson, aged 50, is the eldest son of a former Bishop of Chelmsford, and was educated at Cheltenham and Corpus Christi, Cambridge.

● First scholarships to be awarded by ICI (India) Private Ltd. under a scheme to give advanced technical training in the UK to ten Indian students during the next five years, have been gained by MR. SUBIR CHOWDHURY, B.Sc., and MR. U. D. KINI, B.Sc., of the Institute of Technology, Kharagpur. They are to study mechanical engineering for three years at the Manchester College of Science and Technology.

● MR. PETER BARSBY, chief engineer of Rockweld Ltd., is visiting Canada to organise and schedule production methods and to introduce the latest techniques developed at Croydon in the factory of a subsidiary, Canadian Rockweld Ltd., Toronto. Later, Mr. Barsby is to visit the US to study production methods there.

● MR. S. HOPTON has resigned his position as director and general manager of Stockdale Engineering Ltd., of Poynton, in order to launch a new enterprise, Chemical Equipment Engineering Co., of Stanley Hall Chambers, Castle Street, Macclesfield.

● MR. F. L. COLE has been appointed managing director of the Bartrev Board Co., International Plastics, British Artificial Resin Co. and Plastics Spray in succession to MR. W. J. FISCHBEIN, who has resigned to concentrate on his other business interests.

● SIR PERCY SILLITOE, former head of MI5, and SIR CLINTON PELHAM have joined the board of the Industrial Exchange Co., of which Mr. W. J. Fischbein is chairman and managing director. Sir Percy Sillitoe is shortly to tour Africa to investigate the possibilities of installing chemical plants in certain areas, while Sir Clinton Pelham is to visit Australia, New Zealand, India and Pakistan.

● THE HON. ALEXANDER HOOD, a director of J. Henry Schroder and Co. Ltd., merchant bankers, has joined the board of Petrofina (Gt. Britain) Ltd.

● MR. N. C. LAKE, formerly managing director of the Head Wrightson Machine Co. Ltd, has been appointed deputy managing director of the Head, Wrightson group parent company.

● MR. E. R. BLANE was presented with a motor lawn mower and a tankard to mark the end of his service with Mobil Oil as consultant in the technical service laboratory. Mr. Blane has been responsible for much original work on process products, particularly emulsions, tanners' products and cutting and rolling oils.

Mr. E. R. Blane

● MR. C. A. ADAMS, chief of trials and safety of the UK Atomic Energy Authority Weapons Group at Aldermaston, has been appointed chief nuclear health and safety officer of the Central Electricity Generating Board.

● MR. J. A. JACKSON has resigned from the board of William Blythe and Co. Ltd.

● DR. C. W. CHRISTENSEN, director of microbiology, Difco Laboratories, US, who has been touring Europe since 6 July, is to arrive in London early in September to spend a fortnight visiting institutes in England and Scotland. He also plans to attend the Institute of Medical Laboratory Technology Conference at Bristol, 15 to 18 September.

Thirty members of the Royal Institute of Chemistry's London section recently visited the Crawley works of The APV Co. Ltd. Here they are seen being welcomed by Dr. R. Seligman, the company's president

Commercial News

- **Nearly Two-thirds ICI Loan Stock Converted**
- **Anchor Chemical Maintain Interim at 5%**
- **Offer Received for Anglo-French Shares**
- **Increased Capitalisation of 3 Reichhold Cos.**

Imperial Chemical Industries

Holders of nearly two-thirds of Imperial Chemical Industries' £40 million convertible loan stock have taken the opportunity to convert into ordinary shares. During July £24.3 million loan stock was converted into 17.5 million £1 ordinary shares. Conversion was made on the revised basis announced in May of 36 ordinary shares for every £50 loan stock. The next opportunity for conversion will be in July 1959, when 34½ shares will be offered for each £50 stock. The following year 33 shares for each £50 stock will be offered.

Over 90,000 employees of ICI have qualified for bonus in respect of 1957 under the company's profit sharing scheme. This bonus amounts to £4,993,000 gross (average per employee of £54 17s 6d) and after tax to £3,716,000 (average £40 16s 9d). This year £1,987,000 ordinary stock will be handed over to just over 30,000 employees.

Reichhold Chemicals

Capitalisation of three Reichhold chemical companies in Europe will be increased substantially, making possible major plant expansions for increased and more diversified production of basic chemicals and synthetic resins, Mr. Henry H. Reichhold, president of Reichhold Chemicals, Inc., disclosed this week. This firm has substantial interest in all Reichhold companies overseas. The price of the US stock has approximately doubled since it was admitted to over-the-counter trading 21 months ago.

Capitalisation of Reichhold's Swiss company, Oel and Chemie Werk AG, will be doubled from S. Frs.2.5 to 5 million, making possible construction of a new plant which will produce 3,000 tons of phthalic anhydride per year. Reichhold Chemie AG, in Germany, will double its phthalic capacity following an increase in capitalisation from DM4 to 5 million. Capitalisation of the French firm, Reichhold-Beckacite SA, will rise from Frs.400 to 600 million as capacity for production of various chemicals is increased. Headquarters for the French company will be in Bezons, Paris.

Mr. Reichhold said most of the expansions will be financed by sale abroad of stock in the individual companies. A large part of the proceeds, plus working capital, from sale of the US company's stock are being used for expansion here.

Anglo-French Phosphate

Stockholders in Anglo-French Phosphate Ltd. have now received a formal offer from Robert Benson, Lonsdale and Co. Ltd, of 15s 3d for each of the 750,000 ordinary 5s units.

Unilever, who hold 55.9 per cent of the Anglo-French capital, have agreed to accept. Chairman of the company, Mr. F. J. Pedlar, has stated that it is the intention of the bidders that Anglo-French should dispose of the major part of their holdings in Compagnie Francaise des Phosphates del'Oceanic as soon as possible. Later, other assets will be converted into cash.

Head, Wrightson and Co. Ltd.

After more than 25 years of steadily-rising profits, Head, Wrightson and Co report a record gain of £1,757,581, a net figure of £722,574 after depreciation and taxation. During the year the £1 ordinary shares were divided into 5s. units, and £590,625 of reserves were capitalised and distributed in the proportion of two 5s. shares for every four of the new 5s. shares held. On this reorganised capital an interim of 5 per cent had been paid, and a final of 12½ per cent was recommended, making a total for the year of 17½ per cent (last year 15 per cent) which was covered nearly four times by the group's earnings.

With extensive and increasingly well-equipped research facilities, the group was now able to devote more attention to development work, especially in connection with plant for the iron and steel industry. As a result, the company was in a position to offer complete plants for the manufacture of steel by any of the latest pneumatic processes, such as oxy-Bessemer, Linz-Donawitz, or Kaldo.

Hickson and Welch (Holdings)

The board of Hickson and Welch (Holdings) Ltd., are to seek authority to increase the authorised share capital from £950,000 to £1,400,000 by the creation of 900,000 additional ordinary shares of 10s. each, 600,000 of which would be paid for out of reserves and distributed to shareholders in the proportion of three new shares to every seven ordinary shares held. (See CHEMICAL AGE last week, p. 199.)

Industrial Exchange Co.

The Industrial Exchange Co. announces that Sir Percy Sillitoe and Sir Clinton Pelham have joined the board to assist the chairman and managing director Mr. W. J. Fischbein, in a project to supply chemical and petrochemical plants to Commonwealth countries on an extended credit basis. (See page 235.)

Mr. Fischbein states that the company was formed for the sale and finance of engineering services and equipment for overseas industrial development, and would in addition provide 'know-how' and technical services up to the production stage. Present plans involved the erection of about 15 plants ranging in cost from £500,000 to £4 million.

Capital of about £30 million is to be provided for these projects largely from Swiss, Italian, French, Belgian and UK sources.

Anchor Chemical Co.

Interim dividend of Anchor Chemical Co. Ltd., is 5 per cent (same). Total dividend for last year was 17½ per cent.

IBE

Some improvement in the activities of the IBE group, manufacturers of bitumen emulsions, since the close of the financial year is noted by the chairman, Mr. R. Montgomerie, who suggests that the future may be faced with confidence. Special road emulsions were making a considerable contribution to sales.

Trading profits and sundry income for the year ending 31 March 1958, declined from £132,389 to £101,776, but the directors felt justified in maintaining the dividend at 10 per cent in view of the provisions made in previous years and the improvement in the liquid position.

Inco of Canada

International Nickel of Canada have declared the usual quarterly dividend of 65 cents a share. The total dividend for 1957 was brought up to $3.75 by an additional year-end payment of $1.15 per share.

Johnson, Matthey and Co.

A forecast that the results for the current year would fall noticeably below those for the year ending 31 March 1958, which showed, at £774,021, an increased profit of £48,704, was made by Mr. G. C. H. Matthey, chairman of Johnson, Matthey and Co. Ltd., in his annual report.

The market price of platinum had weakened appreciably since the second half of 1957, principally as a result of changes in the techniques of oil refining. Such improvements had been made in the performance of platinum-bearing catalysts that the requirements of new platinum had been very greatly scaled down, and for the first time for many years the market was over supplied. The refining, fabrication and

marketing of the platinum group metals was so substantial a part of the company's total business that the contraction of the work in this field would have a marked effect on current earnings.

The new plant at Royston to which the company was transferring all its precious metal refining and chemical work was practically complete. The sections already in operation were the gold, silver and platinum refineries, the platinum forge, the analytical laboratories, the plating department, and maintenance, despatch and office services. Sections yet to be transferred were the units producing spectrographically-pure materials, liquid gold, salts and chemical compounds, but the whole project should be completed by the autumn.

A final dividend of 7 per cent bringing the total up to 10 per cent for the year was recommended.

Borax (Holdings)

Borax (Holdings) Ltd., have declared an interim dividend of 1.75d per 5s unit of deferred ordinary stock (subject to income tax) for the year ending 30 September.

Yorkshire Copper

Net profits of Yorkshire Copper Works (Holdings) Ltd., for the half-year ending 27 January 1958 (prior to the start of the merger with the metals division of ICI) were £191,979, as against £420,117 for the 12 months ending 31 July 1957. The interim dividend was maintained at 6 per cent.

Market Reports

Quiet Trading

LONDON Trading conditions on the industrial chemicals market remain steady with the demand on home account reasonably good for the period. Contract deliveries are being taken up according to schedule, and interest in new forward business is expected to show some signs of revival in the coming weeks.

Prices on the whole are steady with no definite trend. Export trade, although less active than a year ago, has been maintained at a good level with Australia the chief outlet.

The market for the coal tar products is quiet and uneventful, but creosote oil, cresylic acid and the xylols are reported to be in active request.

MANCHESTER The past week has again been extremely quiet on the Manchester market for chemicals and allied products. Contract deliveries of the soda compounds and other bread-and-butter lines have been on a smaller scale and there has been a noticeable decline in both the number of fresh inquiries and the volume of new business. This falling off has been largely due to the fact that the holiday season has reached its peak and from now on a gradual improvement in trading conditions should be experienced.

GLASGOW The impact of the holiday period was apparent during the past week in the Scottish heavy chemical market. Conditions were very quiet except as reported last week from those areas not so affected by holidays; however, a resumption to more normal conditions is anticipated for next week. Prices more or less remained unchanged. The demand for agricultural chemicals still remains fairly brisk, while the export market continues to be satisfactory.

TRADE NOTES

Monsanto's Spraying Device

Monsanto Chemicals are featuring Syton W20 and a new spraying device on their stand at the International Textile Machinery and Accessories Exhibition at Belle Vue, Manchester, from 15 to 25 October.

Syton W20, first developed as a spinning assistant to increase yarn strength and improve uniformity on woollen, worsted and modified systems, is also being put forward as an anti-soiling agent for carpets. The new spraying device is designed for use with the Rapier auto-leveller and similar equipment to give an even application of emulsion.

Hoechst Chemicals

Hoechst Chemicals Ltd., 50 Jermyn Street, London SW1, are to open a branch office in Birmingham on 18 August in the charge of Mr. A. L. Welby from the Manchester office. The address will be 115-117 Colmore Row, Birmingham, and the telephone number Central 6046.

German Quartz Agency

Fleischmann (London) Ltd., of Chancery House, Chancery Lane, London WC2, are the sole authorised agents and distributors for the UK of all quartz products manufactured by Heraeus Quarzschmelze G.m.b.H., Hanau, Western Germany, with the exception of optical quartz.

Dust Measuring Instrument

A system for sampling exhaust gases from industrial plants for fumes and dust is being made available commercially by Longworth Scientific Instrument Co., Thames Street, Abingdon, Berks. The unit can be operated by a single man and may also be used for control purposes.

The gas is sampled by means of a stainless steel probe which faces into the stream of flowing gas. The gas collected is fed into a filter unit (containing a filter composed of graded glass fibres) which may be easily detached and weighed. The device, originally developed by the British Iron and Steel Research Association, is said to be able to deal with iron oxide fumes from corrosive gases at 300°C at the rate of 3 cu. ft. per minute.

Beckman Equipment

A. Gallenkamp and Co. Ltd., Sun Street, London EC2, have been appointed sole agents for the UK, as from 1 July 1958, for the range of continuous recording process instruments manufactured by Beckman Instruments Inc. These include their electrolytic hygrometer, pH meters, gas chromatographs, leak detectors, oxygen analysers and flow colorimeters. This is in addition to the agency already held for spectrophotometers, electrophoresis apparatus, CO_2 analysers, gas chromatographs and other laboratory instruments.

Concrete Floor Paint

Allweather Paints Ltd., 36 Gt. Queen Street, London WC2, announce the marketing of a new heavy duty concrete floor paint to be known as Pitacrete. Claimed to be resistant to weak acids and alkalis, petrol splashes and mineral oils, it is quick drying and washable with soaps and detergents.

Grants to Leeds University

Leeds University has received £500 from the Carborundum Co. Ltd. towards research work on refractory materials at the Houldsworth School of Applied Science, and £5,400 a year for five years from Imperial Chemicals for ICI fellowships.

Clean Air Act Guide

A manual to aid works managers and engineers has been produced by the Federation of British Industries under the title 'A Guide for Industry to the Clean Air Act 1956'. This Act came into full operation on 1 June this year and the new publication brings up to date the previous booklet brought out by the Federation. Advice on how to combat smoke, grit and dust is included, and appendices cover such matters as the use of Ringelmann charts and smoke density indicators. The names of manufacturers of various types of equipment are given. The handbook costs 4s and may be obtained from the Federation at 21 Tothill Street, London SW1.

BP's Grangemouth Plant

BP's Grangemouth refinery is to be opened to the public during Scottish week from 1 to 6 September. Visitors will be shown the working units as well as those under construction which will next year bring the annual throughput up to 3,200,000 tons, compared with the present 2,200,000 tons.

De La Rue

Date for the change of name of Thomas De La Rue and Co. Ltd., to the De La Rue Co. Ltd., is fixed for 14 August. On the same date the security printing division will begin operations as Thomas De La Rue and Co. Ltd., and the Potterton division as Thomas Potterton Ltd.

NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London WC2, price 3s 3d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 10 September

Silicone rubber gum composition. Connecticut Hard Rubber Co. **801 134**

Production of aromatic hydroperoxides carbinols, ketones and carboxylic acids. Ruhrchemie AG. **801 387**

Apparatus for separating fine particles from gases. Bigwood & Son, Ltd., J. **801 172, 801 173**

Emulsions. Unilever, Ltd. [Divided out of 801 118.] **801 119**

Method of contacting solid particles with fluids. National Research Council. **801 315**

Process for the production of a cationic surface-active additive for bitumen together with piperazine and ethylenediamine. Swan & Co., Ltd., T. **801 389**

Producing defluorinated phosphate material. Internationale Minerals & Chemical Corp. **801 129**

Production of sterols. Vitamins, Ltd. **801 390**

6-Methyl steroid compounds. British Drug Houses, Ltd. **801 201**

Bacterial digestion of organic matter. Dorr-Oliver, Inc. **801 144**

Process for isobutane-olefin alkylation. Texaco Development Corp. **801 145**

Manufacture of N-tert.-actylbenzthiazl-2-sulphenamides. Imperial Chemical Industries, Ltd. **801 392**

1:3:4-Thiadiazoles and their preparation. British Drug Houses, Ltd. [Cognate application 34193.] **801 394**

Production of ammonium salts of aromatic carboxylic acid. Distillers Co., Ltd. **801 125**

Perfluorochloroolefinic polymers. Minnesota Mining & Manufacturing Co. **801 398**

Method of enlarging solid cross-linked polymer bodies. Dow Chemical Co. **801 399**

Separating components of oleaginous mixtures by fractional crystallisation. International Research & Development Corp. **802 102**

Polymerisation process. Distillers Co., Ltd. [Cognate applications 2676, 3144, and 4945.] **801 401**

Production of iron oxide and sulphur. Chemical Construction Corp. **801 403**

Method of vulcanising rubber. Goodyear Tire & Rubber Co. **801 105**

Polyethylene films and method of making same. Union Carbide Corp. **801 256**

Insolubilising artificial filaments, threads, fibres and the like obtained by the spinning of solutions of proteins. Imperial Chemical Industries, Ltd. **801 324**

Aromatic polyester compositions. Goodyear Tire & Rubber Co. **801 065**

Production of stable solid water-soluble aminoplastic condensation products. Svenska Oljeslageri Aktiebolaget. **801 404**

Liquid compositions useful as a base material for lubricating oils, lubricating grease and hydraulic liquids. Byman, N. R. M. **801 154**

Substituted anilides. Smith, A. E. W. **801 405**

Production of terephthalic and isophthalic acids. Distillers Co., Ltd. **801 126**

Preparation of aromatic compounds. Minnesota Mining & Manufacturing Co. **801 233**

Electrolytic process for the manufacture of aliphatic dinitriles. Goodyear Tire & Rubber Co. **801 406**

Polymerisation process and catalyst therefor. Esso Research & Engineering Co. **801 407**

Process of removing colour from acrylonitrile polymerisation products. American Cyanamid Co. **801 237**

Preparation of fluorocarbon compounds. Du Pont de Nemours & Co., E. I. **801 408**

Filler materials for rubber mixtures, rubber mixtures and products thereof. Soc. Argirec. **801 107**

6-Methyl steroid compounds. British Drug Houses, Ltd. [Divided out of 801 201.] **801 202**

Liquids-granular solids contacting. Socony Mobil Oil Co., Inc. [Addition to 773 421.] **801 409**

Polymer compositions and articles. Du Pont de Nemours & Co., E. I. **801 326**

Method for treating molten metal. Stora Kopparbergs Bergslags Aktiebolag. **801 156**

Properties of aqueous emulsions and dispersions. Badische Anilin- & Soda-Fabrik AG. **801 411**

Preparation of polyalkylene terephthalates. Gevaert Photo-Producten NV. **801 412**

Vinyl imidazimidazole derivative and its polymers. Monsanto Canada, Ltd. **801 413**

Hydroforming process. Esso Research & Engineering Co. **801 086**

Petroleum resin process. Esso Research & Engineering Co. **801 414**

Production of titanum tetraiodide. Monsanto Chemical Co. **801 415**

Fuel for nuclear reactors and process of making same. Abraham, B. M., and Flotow, H. E. **801 288**

Producing unsaturated primary alcohols more particularly those of vitamin A series. Philips Gloeilampenfabrieken NV. **801 416**

Methods of producing unsaturated primary or secondary alcohols. Philips Gloeilampenfabrieken NV. **801 417**

Improving the lubricating properties of hydrocarbon lubricating oils. Shell Research, Ltd. **801 418**

Aluminate glass. Zeiss-Stiftung, C. [trading as Jenaer Glaswerk Schott & Gen]. **801 163**

Impermeable polyethylene film and containers and process of making same. Shulton, Inc. **801 419**

Polyurethane foam products and process of preparation thereof. Goodrich Co., B. F. **801 070**

Method of reducing the halogen content of halohydrocarbons. Abbey, A. (Dow Chemical Co.). **801 127**

Dehalohydrogenation of halohydrocarbons. Abbey, A. (Dow Chemical Co.). [Addition to 801 127.] **801 128**

Copolymers of acrylonitrile and alkenylaromatic sulphonates, and dyeable fibres thereof. Dow Chemical Co. **801 420**

Steroids and the manufacture thereof. Upjohn Co. **801 422**

Phenthiazine derivatives. Soc. des Usines Chimiques Rhone-Poulenc. [Addition to 800 635.] **801 423**

Processing an aqueous phosphorus emulsion. Knapsack-Griesheim AG. **801 246**

Regeneration of catalyst. Esso Research & Engineering Co. **801 090**

Isomerisation process and catalyst therefor. Standard Oil Co. **801 166**

Liquid-coating compositions. Du Pont de Nemours & Co., E. I. **801 337**

Explosive mixtures containing aluminium and in the methods of manufacturing such mixtures. Magnasca, Ltd. **801 193**

Manufacture of titanium tetrachloride. Columbia-Southern Chemical Corp. **801 424**

Production of artificial filaments and the like by the viscose spinning process. Vereinigte Glanzstoff-Fabriken AG. **801 339**

Soluble epinochrome derivatives and method for preparing same. Tanabe Seiyaku Co., Ltd. **801 425**

Curing semi-fluid polychloroprene with polyamines. Du Pont de Nemours & Co., E. I. **801 426**

Pelleted polychloroprene. Du Pont de Nemours & Co., E. I. **801 427**

Solvent dewaxing of petroleum oils. Esso Research & Engineering Co. **801 248**

Protection of ferrous metal parts against liquid molten aluminium. Metallwerk Plansee G.m.b.H. **801 115**

Desulphurisation of hydrocarbon oil and catalyst therefor. Standard Oil Co. **801 428**

Preparation of 2 (alpha-aminoethyl)-furan and derivatives thereof. Olin Mathieson Chemical Corp. **801 169**

Open to public inspection 17 September

Separation of plutonium. Potratz, H. A. **801 520**

Separation of uranium, plutonium and fission products. Spence, R., and Lister, M. W. **801 521**

Fumigating compositions. Murphy Chemical Co., Ltd., and Walpole, J. L. **801 659**

Production of layers by settling materials from liquid, suspensions. Electric & Musical Industries, Ltd. **801 752**

Drier for continuously moving strip material. Kodak, Ltd. **801 754**

Manufacture of pigment dispersions. Farbwerke Hoechst AG. **801 522**

Process for hardening artificial hides or leathers. Alegre, A. A. **801 433**

Alpha-phenyl, alpha-(4-piperidyl)-benzyl alcohol and the preparation thereof. Merrell Co., W. S. **801 712**

Method and apparatus for depositing metal coatings on metal bases. Alexander, P. **801 589**

Production of benzene dicarboxylic acids. Newby, H. (Chemische Werke Hüls AG). **801 562**

Alkaloid and a process for its isolation. Ciba Ltd. [Addition 734 108.] **801 541**

Dehydrogenation of dehydrocarbons. Foster Wheeler, Ltd. **801 563**

Process for treating clays. Fullers' Earth Union, Ltd. **801 722**

Treating fibres, articles made therefrom or films to reduce their tendency to accumulate static charges of electricity. American Cyanamid Co. [Addition to 723 580.] **801 721**

Production of homogeneous multi-component liquid mixtures, solutions or emulsions. Siemens-Schuckertwerke AG. **801 558**

Solvent extraction of neptunium. UK Atomic Energy Authority. **801 742**

Extraction of plutonium and uranium. UK Atomic Energy Authority. **801 743**

Low-temperature carbonisation and gasification of coal in a gas producer. Kohlenscheidungs-G.m.b.H. **801 714**

Purification of glycols by distillation. Imperial Chemical Industries, Ltd. [Cognate application 7956.] **801 723**

Vertical retorts. Simon-Carves, Ltd. (Otto & Co. G.m.b.H., C.). **801 592**

Preparation of activated alumina. Pechiney Compagnie de Produits Chimiques et Electrometallurgiques. **801 462**

Sonic gas analysis apparatus. UK Atomic Energy Authority. **801 757**

Production of vinyl chloride from 1, 2-dichlorethane and acetylene. Soc. Belge de L'Azote et des Produits Chimiques du Marly. **801 663**

Production of yellow vat dyestuffs of the anthraquinone series. Farbenfabriken Bayer AG. **801 687**

Annealing steel in a vacuum. Steirische Gusstahlwerke AG. **801 463**

Phosphoric and thionophosphoric acid esters and a process for their production. Farbenfabriken Bayer AG. **801 523**

Removal of condensible impurity from hydrogen. Stamicarbon N.V. **801 691**

Phthalocyanine and like colouring materials. Bergwerksverband Zur Verwertung von Schutzrechten der Kohlentechnik G.m.b.H. **801 488**

High-purity dimethyl terephthalate and process for its preparation. Mid-Century Corp. **801 567**

Polymerisation of vinyl-aromatic compounds. Petrochemicals, Ltd. **801 555**

Procedure for producing neutral tetracycline from chlortetracycline. American Cyanamid Co. **801 544**

Production of organic compounds containing phosphorus and sulphur. Albright & Wilson (Mfg.), Ltd. **801 568**

Diethylphenylacetic acid esters. British Drug Houses, Ltd. **801 725**

Process for the treatment of glauber's salt. Courtaulds, Ltd. [Cognate application 21548.] **801 527**

Pharmaceutical preparations. Rabinovitch, H. **801 726**

Heat-exchange systems and compositions for cleaning such systems. Smith Bros. & Co. (Chemicals), Ltd., and Kelly, E. J. **801 759**

Reflux condensers. Soc. L'Air Liquide, Soc. Anon. Pour L'Etude et L'Exploitation des Procedes G. Claude. **801 493**

Production of compositions which contain linear poly-condensates or polymers, and from which can be produced threads and fibres with reduced electrostatic charging. Vereinigte Glanzstoff-Fabriken AG. [Addition to 796 800.] **801 727**

Manufacture of potassium pyroantimonate. Associated Lead Manufacturers, Ltd., and Green, W. E. **801 572**

Recovery of thorium. UK Atomic Energy Authority. **801 573**

Manufacture of hydrogen. Esso Research & Engineering Co. **801 671**

Production of glycidyl ethers. Badische Anilin- & Soda-Fabrik AG. **801 728**

Grafting polymers or copolymers. Centre National de la Recherche Scientifique. **801 528**